

AD-A051 800

PRATT AND WHITNEY AIRCRAFT GROUP WEST PALM BEACH FL 6--ETC F/G 21/5
DEVELOPMENT OF ABRASIVE BLADE TIP COATINGS FOR USE IN AN ABRADA--ETC(U)
APR 77 M J WALLACE

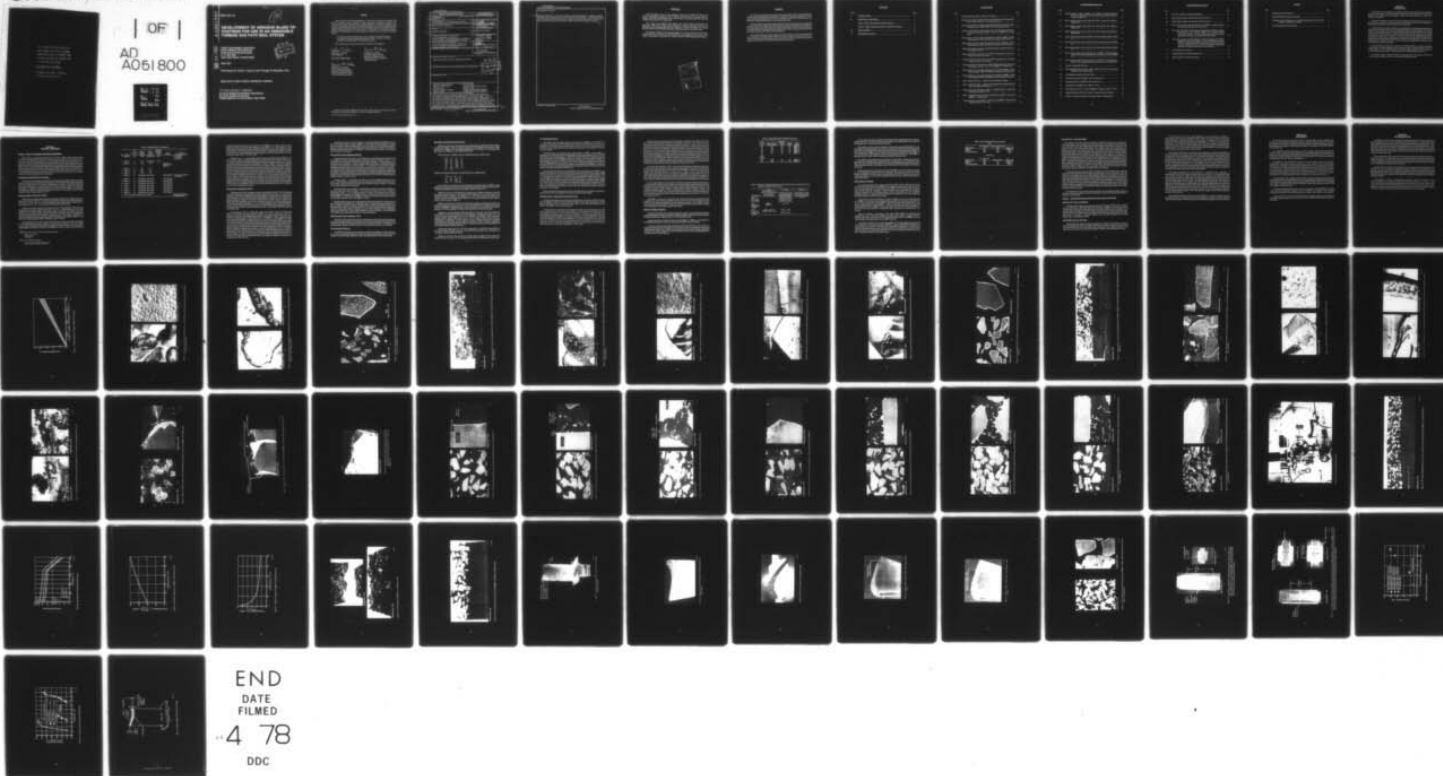
F33615-76-C-5048

UNCLASSIFIED

FR-8324

AFML-TR-77-47

NL



THIS REPORT HAS BEEN DELIMITED
AND CLEARED FOR PUBLIC RELEASE
UNDER DOD DIRECTIVE 5200.20 AND
NO RESTRICTIONS ARE IMPOSED UPON
ITS USE AND DISCLOSURE.

DISTRIBUTION STATEMENT A

APPROVED FOR PUBLIC RELEASE;
DISTRIBUTION UNLIMITED.

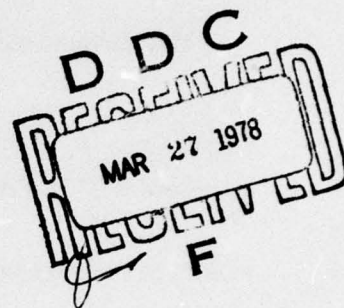
AD A 051800

AFML-TR-77-47

12 SC

DEVELOPMENT OF ABRASIVE BLADE TIP COATINGS FOR USE IN AN ABRADABLE TURBINE GAS PATH SEAL SYSTEM

United Technologies Corporation
Pratt & Whitney Aircraft Group
Government Products Division
P. O. Box 2691
West Palm Beach, Florida 33402



April 1977

Final Report for Period 1 January 1976 Through 31 December 1976

Approved for public release; distribution unlimited

AIR FORCE MATERIALS LABORATORY
Air Force Wright Aeronautical Laboratories
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio 45433

DDC FILE COPY

NOTICE

When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

This report has been reviewed by the Information Office (OI) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nations.

This technical report has been reviewed and is approved for publication.

William T. O'Hara

WILLIAM T. O'HARA
Project Engineer

FOR THE COMMANDER

Norman M. Geyer

NORMAN M. GEYER
Technical Manager for High
Temperature Materials Group
Processing and High Temperature
Materials Branch

Norman M. Tallan

NORMAN M. TALLAN
Chief, Processing and High
Temperature Materials Branch
Metals and Ceramics Division
Air Force Materials Laboratory

Copies of this report should not be returned unless return is required by security considerations, contractual obligations, or notice on a specific document.

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER AFML-TR-77-47	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) DEVELOPMENT OF ABRASIVE BLADE TIP COATINGS FOR USE IN AN ABRADABLE TURBINE GAS PATH SEAL SYSTEM.		5. TYPE OF REPORT & PERIOD COVERED Final Report, 1 Jan 1976 to 31 Dec 1976,
7. AUTHOR(s) Matthew J. Wallace		6. PERFORMING ORG. REPORT NUMBER FR-8324
9. PERFORMING ORGANIZATION NAME AND ADDRESS United Technologies Corporation Pratt & Whitney Aircraft Government Products Div P. O. Box 7691, West Palm Beach, FL 33402		8. CONTRACT OR GRANT NUMBER(s) F33615-76-C-5048
11. CONTROLLING OFFICE NAME AND ADDRESS Air Force Materials Laboratory (AFML/LLM) Air Force Wright Aeronautical Laboratories Wright-Patterson AFB OH 45433		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 62120F, 7312, 731201 73120140
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		12. REPORT DATE April 1977
		13. NUMBER OF PAGES 69
		15. SECURITY CLASS. (of this report) UNCLASSIFIED
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited.		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Aluminum Oxide Coatings Rub Tolerance Blade Tip Treatment Silicon Carbide Abrasive Grits Ceramic Seals Sputter Coated Diffusion Barrier Coatings Transient Liquid Phase Bonding Outer Air Seals Vacuum Hot Pressed Compacts		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) An abrasive blade tip treatment coating was developed which exhibited promising rub resistance against ceramic (graded ZrO_2 -Ni Cr) outer air seals. This blade/seal system was designed for advanced high pressure turbine sealing applications. The best tip treatment was produced by vacuum hot pressing a compact comprised of 50 volume percent of coated 8-mil diameter silicon carbide (SiC) grits in a MERL 711 (CoNiCrAlY) alloy matrix. The SiC grits were radio frequency (RF) sputter coated with a 0.1-mil thick aluminum oxide diffusion barrier coating prior to insertion in the matrix. This compact was transient liquid phase bonded to prepared rig		

DD FORM 1 JAN 73 1473

EDITION OF 1 NOV 65 IS OBSOLETE
S/N 0102-LF-014-6601

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

DDC
RECEIVED
MAR 27 1978
F

392 887

TC

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE(When Data Entered)

Continuation of Item 20.

→ and engine blade tips. The abrasive blade tip withstood fabrication, bonding, and heat treatment cycles with little or no grit dissolution and degradation. Blade/seal dynamic rub tests produced little blade tip wear (compared with seal wear) with no dynamic rub impact problems or any loss of abrasive blade tip treatment structural integrity. ↑

S/N 0102-I.F-014-6601

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE(When Data Entered)

FOREWORD

This Final Report covers all work performed under Contract F33615-76-C-5048 by the United Technologies Corporation, Pratt & Whitney Aircraft Group, Government Products Division, West Palm Beach, Florida, from 1 January 1976 to 31 December 1976. The report was released by the author on 31 January 1977.

This contract was initiated under Project 7312, "Metal Surface Deterioration and Protection," Task 731201, "Metal Surface Protection," Work Unit 73120140. The work was performed under the technical direction of Mr. William T. O'Hara of the Metals and Ceramics Division of the Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio.

Mr. Matthew J. Wallace was the Program Manager for the Pratt & Whitney Aircraft Group, Government Products Division, and was responsible for the management and execution of the program. Appreciation is extended to Mr. Gerald A. Majocha, P&WA Experimental Engineer.

ACCESSION for	
NTIS	White Section <input checked="" type="checkbox"/>
DDC	Buff Section <input type="checkbox"/>
UNANNOUNCED	
JUSTIFICATION	
BY	DISTRIBUTION/AVAILABILITY CODES
	SPECIAL
A	

SUMMARY

An abrasive blade tip treatment coating was developed under Air Force Contract F33615-76-C-5048 that exhibited promising rub resistance in conjunction with ceramic (graded ZrO_2 - NiCr) outer air seals as developed under Navy Contract N00140-74-C-0586. This blade/seal system was designed for advanced high pressure turbine sealing application.

The selected best tip treatment was produced by vacuum hot pressing a compact comprised of 50 vol% 8 mil silicon carbide (SiC) grits coated with aluminum oxide (Al_2O_3) in a MERL 711 (CoNiCrAl) alloy matrix. This compact was transient liquid phase (TLP[®]) bonded to prepared rig and engine blade tips. The abrasive blade tip withstood fabrication, bonding, and heat-treatment cycles with minimum grit dissolution and degradation.

The abrasive blade tip provided excellent rub tolerance results. A chemical etching process was incorporated to eliminate matrix material from the tip surface which resulted in extensive ceramic seal wear and minimal blade tip wear and blade tip transfer. Structural integrity of the compact and bond areas was maintained during the rub interactions.

CONTENTS

Section		Page
I	INTRODUCTION.....	1
II	TECHNICAL DISCUSSION.....	2
	Phase I - Blade Tip Material/Process Development.....	2
	Phase II - Engine Simulation Evaluation of Blade Tip Coating.....	12
III	CONCLUSIONS.....	14
IV	RECOMMENDATIONS.....	15

ILLUSTRATIONS

Figure		Page
1	Turbine Efficiency Penalty vs Blade Tip Clearance.....	16
2	Alumina Coated Silicon Carbide Grains As-Coated From the RF Sputtering Run at 1200°F, 600w, 2-in. Dia. Target for 95 Hr.....	17
3	Alumina Coated Silicon Carbide Grains Mechanically Fractured From the RF Sputtering Run at 1200°F, 600w, 2-in. Dia. Target for 95 Hr.....	18
4	Alumina Coated Silicon Carbide Grains From the RF Sputtering Run As-Vacuum Hot Pressed at 2100°F, 1 Hr, 5 ksi in MERL 711 Co Base Alloy.....	19
5	Thirty Percent by Volume Al ₂ O ₃ Sputtered SiC at 1200°F in MERL 711 After TLP® Bonding to PWA 1422 Blade and Solution Heat Treatment....	20
6	Thirty Percent by Volume Al ₂ O ₃ Sputtered SiC at 1200°F in MERL 711 After TLP® Bonding to PWA 1422 Blade and Solution Heat Treatment....	21
7	Alumina Coated SiC Grits From the RF Sputtering Run at 1600°F, 600w, 2-in. Dia. Target for 94 Hr.....	22
8	Alumina Coated SiC Grits From the 1600°F Sputtering Run Fractured to Show the Coating Cross Section.....	23
9	Alumina Coated SiC Grits From the 1600°F Sputtering Run Showing a Geometry-Related Coating Defect.....	24
10	Alumina Coated SiC Grits From the 1600°F RF Sputtering Run as Vacuum Hot Pressed at 2100°F, 1 Hr, 5 ksi in MERL 711 Co Base Alloy.....	25
11	Thirty Percent by Volume Al ₂ O ₃ Sputtered SiC at 1600°F in MERL 711 After TLP® Bonding to PWA 1422 Blade and Solution Heat Treatment....	26
12	Thirty Percent by Volume Al ₂ O ₃ Sputtered SiC at 1600°F in MERL 711 After TLP® Bonding to PWA 1422 Blade and Solution Heat Treatment....	27
13	HfC+C Coated SiC Grits — Chemical Vapor Deposition Coating.....	28
14	HfC+C Coated SiC Grits — Chemical Vapor Deposition Coating Fractured to Show the Coating Cross Section.....	29
15	Scanning Electron Microscope Image of Oxidized HfC+C Coated SiC Showing Spotty HfO ₂ on SiC.....	30
16	HfC+C Coated SiC Grits as Vacuum Hot Pressed at 2100°F, 1 Hr, 5 ksi in MERL 711 Co Base Alloy.....	31
17	Thirty Percent by Volume HfC+C Coated SiC and MERL 711 Vacuum Hot Pressed at 2100°F, 1 Hr, 5 ksi.....	32

ILLUSTRATIONS (Continued)

Figure		Page
18	Inverted Image of Area in Figure 17 as Viewed in Scanning Electron Microscope Showing SiC Grit With Reacted and Unreacted HfC+C Coating in MERL 711.....	33
19	Thirty Volume Percent Al ₂ O ₃ Coated SiC Grits Vacuum Hot Pressed in TIPALLOY I™.....	34
20	Forty Volume Percent Al ₂ O ₃ Coated SiC Grits Vacuum Hot Pressed in TIPALLOY I™.....	35
21	Fifty Volume Percent Al ₂ O ₃ Coated SiC Grits Vacuum Hot Pressed in TIPALLOY I™.....	36
22	Thirty Volume Percent Al ₂ O ₃ Coated SiC Grits Vacuum Hot Pressed in MERL 72.....	37
23	Forty Volume Percent Al ₂ O ₃ Coated SiC Grits Vacuum Hot Pressed in MERL 72.....	38
24	Fifty Volume Percent Al ₂ O ₃ Coated SiC Grits Vacuum Hot Pressed in MERL 72.....	39
25	Forty Volume Percent Al ₂ O ₃ Sputtered SiC in MERL 72 TLP® Bonded at 2000°F, 8 Hr Showing Minimal Reaction Between the SiC and MERL 72.....	40
26	Forty Volume Percent Al ₂ O ₃ Sputtered SiC in TIPALLOY I™ TLP® Bonded at 2000°F, 8 Hr Showing Reaction Between the SiC and TIPALLOY I... ..	41
27	Dynamic Abradability Rub Rig.....	42
28	Rub Tested Rig Blade of 30 Vol % Al ₂ O ₃ Coated SiC Vacuum Hot Pressed in MERL 711 and TLP® Bonded.....	43
29	Hot Hardness of Selected M-CrAlY Alloys.....	44
30	Thermal Conductivity of MERL 711/SiC Abrasive Grit.....	45
31	Thermal Expansion of MERL 711/SiC Abrasive Grit.....	46
32	Stress Rupture of MERL 711 at 1800°F, 11.4 Hr.....	47
33	Stress Rupture of 50 Vol % SiC Grits/MERL 711 Matrix at 1800°F, 0.9 Hr..	48
34	Engine-Size Blade (PWA 1422) Used for Thermal Fatigue Evaluation.....	49
35	Oblique View Showing Abrasive Coating and Bond on Engine Blade.....	50

ILLUSTRATIONS (Continued)

<i>Figure</i>		<i>Page</i>
36	Top View of Abrasive Coated Engine Blade.....	51
37	Engine Blade Compact After 463 Thermal Shock Cycles.....	52
38	Engine Blade Compact After 463 Thermal Shock Cycles.....	53
39	Fifty Volume Percent Al_2O_3 Sputtered SiC in MERL 711 Matrix Showing Good Al_2O_3 Coating and No Grit Reaction.....	54
40	A) Ceramic ($ZrO_2/NiCr$ Graded Structure) Rig Segment Rubbed at Room Temperature by Six SiC/ Al_2O_3 MERL 711 Coated Rig Blades (Chemically Etched), and (B) Corresponding (Typical) Rubbed Blade With Grits Intact and Minimal Matrix Smearing.....	55
41	A) Ceramic Segment Tested at Maximum Observed Temperature of 2800°F Rubbed by Six SiC/ Al_2O_3 MERL 711 Coated Rig Blades (Chemically Etched), (B) Tested Blade Exhibiting Minimum Tip Wear Revealing Grits are Unaffected by Rub, and (C) Tested Blade Tip Exhibiting Maximum Tip Wear and Incipient Matrix Smearing.....	56
42	Rub Evaluation Comparison.....	57
43	Thermal Response of Interacted Blade Tips.....	58
44	Selected Blade Tip Treatment Coating.....	59

TABLES

<i>Table</i>		<i>Page</i>
1	Material/Process Combinations.....	3
2	Single-Bladed Rub Evaluation Summary.....	9
3	Summary of Alloy Performance in Cyclic Hot Corrosion at 1835°F and Cyclic Oxidation at 2000°F and at 2100°F.....	9
4	Stress Rupture/Tensile Test Results.....	11

SECTION I INTRODUCTION

The objective of this program is to develop an abrasive turbine blade-tip coating material and process combination to produce blade tips suitable for operation as part of a first-stage high-pressure turbine (HPT) gas-path abradable ceramic seal system operating at surface temperatures up to 2600°F.

Abrasive tip treatment for turbine blades is an important element along with ceramic static shroud seals for advancing turbine seal system technology to improve the performance of advanced military engines with high turbine-inlet temperatures.

Turbine efficiency is directly related to the maintenance of a tight clearance between the blade tip and the turbine shroud. Loss of tight blade tip/shroud clearance results in loss of high energy air over the blade tips with no work being extracted by the turbine blades and a resultant loss of turbine efficiency (figure 1). Blade tip/shroud clearances are affected by the thermal response of the components, durability of the components in the high-temperature gas turbine environment, and wear of the components as a result of rubbing contact. Thermal response of the components can be controlled by application of cooling air and sophisticated clearance control design schemes. Durability and wear of the blade tip/shroud components is a function of the environment and materials properties.

Pratt & Whitney Aircraft has conducted a two-phase program to develop an abrasive turbine blade tip coating material/process combination under Air Force Contract F33615-76-C-5048, Project No. 7312, suitable for operation as part of a first-stage HPT gas path ceramic seal system developed under NAPTC Contracts N00140-74-C-0586 and N00140-76-C-0971.

The Phase I effort consisted of blade tip material/process development and Phase II involved engine simulation evaluation of the selected blade tip coating.

SECTION II TECHNICAL DISCUSSION

PHASE I - BLADE TIP MATERIAL/PROCESS DEVELOPMENT

Phase I blade tip material/process selections are shown in table 1 and consisted of three M-CrAlY matrix alloys, three SiC grit volume percents, two diffusion barrier materials and processes, and TLP[®] bonding parameters. The best diffusion barrier material and process was used in the evaluation of the vacuum hot pressing (VHP) M-CrAlY matrix alloys and volume percents SiC grits. The TLP bonding parameters were selected for the SiC grit/M-CrAlY matrix VHP compacts to join the PWA 1422 (directionally solidified MarM-200 with Hf) blades used for dynamic rub testing with the ceramic turbine seal developed under Naval Air Propulsion Test Center (NAPTC), Trenton, N.J., Contract N00140-74-C-0586. Wear characteristics, in conjunction with blade tip coating properties, stress rupture, and thermal shock testing, were evaluated to produce Phase II material/process combinations.

Diffusion Barrier Materials and Processes

Aluminum oxide and hafnium oxide (HfO₂) were selected as the diffusion barrier materials in order to minimize or eliminate SiC grit dissolution in the M-CrAlY alloy matrix. Aluminum oxide was chosen based on its predicted thermodynamic stability in M-CrAlY alloys and preliminary experiments with radio frequency (RF) sputtered Al₂O₃ coatings which indicated some degree of dissolution protection. Hafnium oxide was selected based on its predicted thermodynamic stability in M-CrAlY alloy and the ability of the industry to produce HfO₂ coating by chemical vapor deposition (CVD).

Al₂O₃ RF Sputter Coated SiC at 1200°F

Three grams of ultrasonically cleaned 8 mil diameter SiC¹ grits were sputter coated for a total of 95 hr at 1200°F ± 25°F. A 2-in. diameter Al₂O₃ target, 99.995% pure, was RF sputtered at 600 watts to coat the SiC. Pre- and post-sputtering target measurements indicated that a sputtering rate of 2.6×10^{-4} in./hr was achieved during the coating process.

Samples of the Al₂O₃ coated SiC were sputtered with a gold palladium (AuPd) alloy and examined in the scanning electron microscope: (1) in the as-coated condition, and (2) after mechanically fracturing a sample of the grains. Figure 2 illustrates the alumina coated SiC material in the as-coated condition. Figure 3 illustrates the alumina coated SiC material after being mechanically fractured to observe the coating cross section. The alumina coating shows a typical columnar structure with an average coating thickness of approximately 0.00015 in. (0.15 mil), and a thickness range of 0.0002 to 0.00005 in. (0.2 to 0.05 mil).

Two samples of 30% by volume alumina coated SiC were vacuum hot pressed with MERL 711² atomized cobalt alloy powder at 2100°F, 1 hr, and 5 ksi in vacuum (10^{-4} torr). One sample was sectioned, metallographically prepared and examined. Figure 4 illustrates the as-vacuum hot pressed sample of alumina coated SiC in the MERL 711 matrix. In most cases the alumina

¹ Spectrographic analysis for purity yielded the following SiC impurities:

0.01 to 0.1% Al, Fe, Ni, V
0.005 to 0.05% B
<0.01 Mg, Ti

² MERL 711 Lot 6485 chemical analysis:

Bal. Co, 14.5% Ni, 25.1% Cr, 5.4% Al, 5.7% Ta,
0.43% Y, 0.01% C, 0.01% Mn, and 0.07% Si.

Table 1. Material/Process Combinations

No.	Matrix	SiC Grit Concentration (%)	Diffusion Barrier Materials	Diffusion Barrier Processes	Sputtering Substrate Temperature (°F)	CVD Parameters	TLP* Bonding Parameters
1	MERL 711	30	Al ₂ O ₃	RF Sputtering	1200	-	6 hr at 2000°F 4 hr at 2100°F 2 hr at 2200°F
2	MERL 711	30	Al ₂ O ₃	RF Sputtering	1600	-	
3	MERL 711	30	Al ₂ O ₃	CVD	-	Contractor approved vendor definition	
4	MERL 711	30	Al ₂ O ₃	CVD	-		
5	MERL 711	30	HfO ₂	CVD	-		
6	MERL 711	30	HfO ₂	CVD	-		
7	MERL 72	30	Best Material	Best Process	-	Best Parameters	(Temp, time, composition to be defined)
8	MERL 72	40	Best Material	Best Process		Best Parameters	
9	MERL 72	50	Best Material	Best Process		Best Parameters	
10	Tipaloy I	30	Best Material	Best Process		Best Parameters	
11	Tipaloy I	40	Best Material	Best Process		Best Parameters	
12	Tipaloy I	50	Best Material	Best Process		Best Parameters	
13	MERL 711	40	Best Material	Best Process		Best Parameters	
14	MERL 711	50	Best Material	Best Process		Best Parameters	
15	Best blade tip coating material/process combination						Optimize TLP parameters for engine hardware

coating had prevented reaction between the SiC and MERL 711 Co alloy, however, several isolated SiC grains did reveal some dissolution in the MERL 711 Co alloy matrix. A small percentage of the irregularly shaped SiC grains were believed to have geometry-related alumina coating defects which allowed reaction between these SiC grains and the MERL 711 metal matrix. Also, X-ray phase identification was completed on the 1200°F Al₂O₃ sputtered SiC grits. The phases present were: α -SiC and δ -Al₂O₃.

The second vacuum hot press sample of 30% by volume coated SiC-balance MERL 711 was TLP bonded to a sample of blade material PWA 1422 and solution heat treated at 2200°F for 2 hr. Figure 5 illustrates the TLP bonded and solution heat treated sample cross section. A majority of the silicon carbide had dissolved in the MERL 711 matrix. Dissolution of SiC grits appeared throughout the sample, however, not all SiC grits were dissolved. Figure 6 illustrates a dissolved SiC grit between several undissolved grits. Some Al₂O₃ coating can be seen around the outline of the dissolved SiC grit and is also observable around the adjacent undissolved SiC grits. Scanning electron microscopy analysis of the area was performed to identify the constituents and phases present in the dissolved SiC grit area as shown in figure 6B. The major constituents present in the X-ray images of the dissolved SiC grit were Cr and Co; the minor constituents present were Si, Al, Ni, Cr and Co. Both carbon and tantalum appeared, at low concentrations, with a reasonably uniform distribution. The major and minor constituent locations were then isolated at a high magnification and the dispersed X-rays were analyzed; the results confirmed the X-ray image analysis. Further X-ray phase identification of the major constituent area showed the presence of α -Al₂O₃ and Co-Cr solid solution. The minor constituent phase identification was not resolvable due to its small volume fraction present.

Al₂O₃ Sputter Coated SiC at 1600°F

Three grams of 8 mil SiC was sputter coated for a total of 94 hr at 1600°F \pm 25°F. Specimens of the Al₂O₃ coated SiC were examined in the scanning electron microscope: (1) in the as-coated condition, and (2) after mechanically fracturing a sample of the grits. Figure 7 illustrates the Al₂O₃ coated SiC in the as-coated condition. Mechanically fractured grits in the coating cross section are depicted in figure 8. The Al₂O₃ coating revealed a columnar structure similar to the 1200°F RF Al₂O₃ sputtering run but with a somewhat finer grit size. The average coating thickness was approximately 0.00015 in. (0.15 mil) with a thickness range of 0.0002 to 0.0008 in. (0.2 to 0.08 mil), which is essentially the same as the 1200°F RF Al₂O₃ sputtering trial. Thinner coatings, as shown in figure 9, were detected and attributed to grit geometry and/or RF sputtering line-of-sight focus.

Two specimens of 30% by volume Al₂O₃ coated SiC were also vacuum hot pressed with MERL 711 atomized cobalt alloy powder at 2100°F, 1 hr, 5 ksi in vacuum (10⁻⁴ torr). One specimen was sectioned, metallographically prepared, and examined. Figure 10 illustrates the as-vacuum hot pressed sample of Al₂O₃ coated SiC in the MERL 711 matrix. Aluminum oxide coating prevented dissolution of approximately 95% of the SiC grits in the MERL 711 Co base alloy. The second specimen of the 30% by volume SiC grits from the 1600°F RF sputtered Al₂O₃ run was TLP bonded to a sample of PWA 1422 blade material and solution heat treated at 2200°F for 2 hr. Figure 11 illustrates the TLP bonded and solution heat treated sample cross section. Significantly less SiC had dissolved compared to the 1200°F sputtered run. Figure 12 illustrates several completely dissolved grits adjacent to several unattacked grits and two completely unattacked SiC grits. The appearance of the phases in the dissolved SiC grits appears similar to those analyzed in the 1200°F sputtered run. The edges were more affected than the center as shown in figure 5. The center is only approximately 10% affected as compared to almost all of the SiC grits at the edges. The accelerated dissolution at the edges was thought to be caused by the wetting action of the TLP alloy.

An oxidation treatment was then evaluated to reduce the wetting of the MERL 711 matrix by the TLP bonding alloy. A sample of MERL 711 with 1600°F RF sputter coated SiC abrasive grits was oxidized at 2100°F for approximately 1 hr. The surface to be TLP bonded was surface ground to remove the oxide film, TLP bonded, and heat treated for 2 hr at 2200°F. The oxidation treatment eliminated the wetting of the MERL 711 tip by the TLP alloy and 85% of the SiC grits were not affected at the edges.

HfO₂ Chemical Vapor Deposited (CVD) SiC

Numerous attempts were made to deposit HfO₂ on SiC with 100g batches of approximately 8 mil diameter SiC. The repeated attempts consistently resulted in deposition of HfC + C as identified by X-ray diffraction. Abrasive SiC grits coated with HfC + C were examined in the scanning electron microscope: (1) in the as-coated condition and (2) after mechanically fracturing a sample of the grits. Figure 13 reveals the HfC + C coated SiC in the as-coated condition. Mechanically fractured grits within the coating cross section are shown in figure 14. The HfC + C coating had a fine, continuous non-directional structure except for a ridge in the coating which ran parallel to the grit surface about one-third of the way through the coating from the substrate. The ridge in the coating was believed to be the result of two separate coating runs with grits. The average total coating thickness was approximately 0.0003 in. (0.3 mil) with a thickness range of 0.00018 to 0.005 in. (0.18 to 0.5 mil).

A sample of HfC + C coated grits was oxidized in an air atmosphere furnace for 2 hr at 2100°F. X-ray diffraction of the SiC grits from this sample indicated the presence of HfO₂ and β SiC. However, binocular examination of grits from this sample, as shown in figure 15, indicated that the HfO₂ coating was not continuous.

A specimen of 30% by volume HfC + C coated SiC was vacuum hot pressed with MERL 711 atomized cobalt alloy powder at 2100°F, 1 hr, and 5 ksi in vacuum (10^{-4} torr) to assess the effectiveness of HfC + C as a diffusion barrier on the SiC. The specimen was sectioned, metallographically prepared, and examined. Figure 16 illustrates the as-vacuum hot pressed sample of HfC + C coated SiC in the MERL 711 matrix. The coating was only slightly effective in preventing dissolution of SiC in the MERL 711 matrix.

Metallurgical and X-ray image analysis determined that more than 95% of the grits reacted with the matrix alloy, MERL 711. A magnified view of a dissolved grit and unreacted grit is shown in figure 17. Figure 18 displays a scanning electron microscope image from which it was determined that the dissolved SiC area had Co, Cr, Si, and Ni as major element constituents. Also the X-ray image results determined that the coating displayed a non-homogeneous composition of HfC and C. Further, Hf was detected at the grit boundary of the dissolved grits which indicates the ineffectiveness of HfC as a dissolution barrier.

Al₂O₃ Chemical Vapor Deposition on SiC

Trial Al₂O₃ CVD coating runs were made on 100g batches of SiC. After several coating runs no successful coating deposition parameters could be obtained. Samples were examined with a binocular microscope. No coating was observed on the SiC and all facets of the abrasive grits appeared sharp. Therefore, further evaluation of this deposition technique was terminated.

Recommended SiC Barrier

The 1600°F RF sputtered Al₂O₃ coating had revealed the best diffusion coating for SiC grits and was the recommended selection for use in the remaining development of the abrasive tip coating. The chemical vapor deposition of HfO₂ and Al₂O₃ resulted in very limited success.

Alloy Matrix and SiC Grit Volume Percent

Sufficient 8 mil SiC grits coated with Al_2O_3 by RF sputtering at 1600°F were made available for fabrication within vacuum hot pressed compacts of 30, 40, and 50 vol% SiC grit in metal matrices of: (1) MERL 72, and atomized cobalt base alloy, (2) TIPALLOY I™, an atomized nickel base alloy and (3) MERL 711, an atomized cobalt alloy.

Chemical analysis of MERL 72 gave the following results in weight percent:

41.9	Co	0.3	C
25.5	Cr	0.2	Ti
13.7	Ni	0.18	Fe
9.3	W	0.07	Si
4.2	Al	0.02	Zr
3.7	Ta	0.05	Nb
1.6	Hf		

TIPALLOY I atomized powder gave the following results in weight percent:

54.33	Ni	25	Cr
6	Al	8.3	W
5.2	Ta	0.03	Y
0.27	C	0.87	Hf

Rub rig blade tip compacts were then fabricated with matrix alloys of TIPALLOY I and MERL 72 containing 30, 40, and 50 vol% SiC grits coated with Al_2O_3 by RF sputtering at 1600°F. The blade tip compacts were vacuum hot pressed at 2100°F, 5 ksi for 1 hr.

TIPALLOY I with 30 vol% SiC grits shows minimal porosity (figure 19). However, as the SiC grit volume percent increases to 40 (figure 20) and 50 (figure 21) the porosity increases. The TIPALLOY I matrix contained a fine dispersion of γ' and γ phases with some cracks. Also a minimal reaction of the SiC grits with the TIPALLOY I was observed (figures 20 and 21) which is similar to the SiC grits in MERL 711.

MERL 72 with the 30, 40, and 50 vol% of SiC grits are shown in figures 22, 23, and 24. The porosity increases with volume percent SiC as with the TIPALLOY I for comparable SiC volume percents. The MERL 72 matrix contains a γ phase with some cracks. No reaction was observed between the SiC grit and MERL 72 which may be attributed to the greater porosity.

Macroscopic cracks attributed to blade tip porosity were observed in the 40 vol% TIPALLOY I and MERL 72 tips after preparation for TLP bonding. To reduce the tendency for crack formation, several trial samples of the 40 vol% TIPALLOY I and MERL 72 were vacuum hot pressed at 2150°F, 2200°F, 2250°F and 5 ksi for 1 hr in an effort to reduce blade tip porosity. Metallographic examination of these samples did show reduced porosity with increasing temperature. SiC dissolution did not appear to increase at 2150°F or 2200°F compared to previous samples hot pressed at 2100°F. However, the samples vacuum hot pressed at 2250°F did reveal severe reaction and SiC dissolution.

Replacement blade tips of 40 vol% Al_2O_3 sputtered SiC in TIPALLOY I and MERL 72 vacuum hot pressed at 2200°F, 5 ksi for 1 hr were fabricated resulting in the elimination of the macroscopic cracks and reduced porosity.

MERL 711 with 30%, 40%, and 50% by volume Al_2O_3 sputtered SiC were vacuum hot pressed at 2100°F, 5 ksi, and 1 hr. The specimens showed little or no dissolution of the SiC grits.

TLP Bonding Parameters

TLP bond trials were made between metal matrices of MERL 72 and TIPALLOY I containing abrasive grits and PWA 1422 material to select bonding parameters. The trials were conducted at a bond temperature of 2100°F and at times of 4, 8, and 16 hr. Examination of the 2100°F/8 hr trial revealed severe SiC grit dissolution; subsequently a less aggressive TLP bond cycle was pursued.

TLP bond trials were made between metal matrices of MERL 72 and TIPALLOY I containing SiC abrasive grits and PWA 1422 material to select a less stringent TLP cycle than the 2100°F/8 hr cycle. The selected cycle of 2000°F/8 hr showed little if any reaction between the Al_2O_3 sputtered SiC and the MERL 72 (figure 25); however, an extreme reaction between the Al_2O_3 sputtered SiC and the TIPALLOY I was observed (figure 26). No additional reduction in TLP bonding temperature was allowable due to the melting temperature of the TLP foil, therefore, TIPALLOY I was discarded as a candidate matrix material. The MERL 711 specimens, as mentioned previously, exhibited minimal SiC grit dissolution after the TLP bond cycle even after a solution heat treatment at 2200°F for 2 hr.

Rub rig blade fabrication for single-bladed rub tests was completed for the MERL 711 and MERL 72 with 30%, 40%, and 50% by volume Al_2O_3 sputtered SiC abrasive tip compacts. MERL 72 with 30% and 50% by volume Al_2O_3 sputtered SiC were vacuum hot pressed at 2100°F, 5 ksi, 1 hr, machined, pre-oxidized for 1 hr at 2100°F and successfully TLP bonded at 2000°F/8 hr. The 40 vol% abrasive grit compact was vacuum hot pressed at 2200°F to examine degree of matrix porosity reduction at the higher temperature, while other parameters remained constant. MERL 711 with 30%, 40%, and 50% by volume Al_2O_3 sputtered SiC were vacuum hot pressed at 2100°F, 5 ksi, 1 hr, machined, pre-oxidized for 1 hr at 2100°F and TLP bonded to a PWA 1422 rub rig blade. All rig blades were contoured to remove excess tip material and fluorescent penetrant inspected for bond integrity.

Minor bond defects were detected in most of the blades but were considered acceptable for testing based on prior test background with similar minimum defect blades.

Blade/Seal Wear - Single Blade Dynamic Rub Tests

All single blade dynamic rub tests were conducted with the ceramic turbine seal (ZrO_2 -NiCr) developed in NAPTC Contract N00140-74-C-0586. All tests were conducted at a surface speed of 1000 ft/sec and at an interaction rate of 0.001 in./sec. Tests were performed on the dynamic abrasability rub rig shown in figure 27.

An initial rub test was conducted to determine the dynamic structural integrity of the VHP Al_2O_3 coated SiC grits in a M-CrAlY matrix (MERL 711) TLP bonded to PWA 1422 blades with the ceramic seal. The rub test, run at ambient temperature, resulted in 1.5 mil ZrO_2 seal wear with programmed metal transfer to the seal. The blade tip was metallographically examined. Figure 28 illustrates the lengthwise cross section of the blade tip. Approximately 90% of the Al_2O_3 coated SiC exhibited no reaction with the MERL 711 metal matrix material. Porosity in the TLP bond can be observed near one end of the sample, which corresponded to the pretest Zyglo indications; however, no evidence of debonding as a result of the rub testing could be observed at the bond joint or in the 30% SiC grit-MERL 711 VHP compact.

Subsequent tests involved more sophisticated wear investigation and analysis. Analysis of the rub tests completed against MERL 711 and MERL 72 with 30%, 40%, and 50% by volume Al_2O_3 sputtered SiC abrasive tip compacts indicated consistency of results. Seal wear values achieved before blade tip metal transfer ranged from 0.0015-0.003 in. A slight trend for improved seal wear was evident for the higher grit volume percentages for both matrices. However, the true effectiveness of the increased volume percentages of grit was masked by the exposure of matrix material at the tip surface. The matrix material, due to elevated temperatures caused by rubbing friction, began depositing on the ceramic surface at the maximum interference depth of 0.0045 in. Rub-induced time/temperature effects were monitored and revealed high temperature vs time rates in excess of 450°F/sec after initial interference. Pyrometry low-base sensitivity level was measured at 1200°F. Therefore, within a few seconds of interference, the blade tip surface temperature exceeded 2000°F and metal transfer resulted. This transfer phenomena significantly decreased tip treatment effectiveness preventing any additional seal wear.

An iteration was required in order to utilize the abrasive grits more effectively; therefore, a method of chemically etching alloy matrix without affecting the SiC grits and controlling depth of metal removed was developed. The 50 vol% SiC grits in MERL 711 and 72 matrix systems were selected and chemically etched to a depth of 3 to 4 mils at the tip surface. Pretest visual/magnification inspection of the chemically etched MERL 72 matrix blade with 50% by volume Al_2O_3 sputtered SiC abrasive tip compact revealed some chemical reaction further into the compact than programed producing porosity and possible suspect durability. The MERL 711 blade, with the same grit volume percent, did not exhibit any distinguishable surface problems.

The two single-bladed rub tests using the chemical etching process blade tips demonstrated a marked increase in seal wear and the ability to withstand a larger interference before blade tip transfer than the unetched blade tips. Both matrix material blades survived the interaction with no apparent compact integrity problems. Average seal wear values increased to 0.005 in. with maximum interference depth of 0.010 in. before initiation of blade tip transfer occurred. The volume wear factor (VWF) (volume of blade material removed/volume of seal material removed) for the chemically etched blade rub tests was reduced approximately by a factor of 2. Test results are tabulated in table 2.

The MERL 711 matrix with 50 vol% SiC grits with chemically etched blade tips exhibited comparable wear characteristics to MERL 72 matrix with 50 vol% SiC grits chemically etched blade tips but without the additional porosity problems produced in the MERL 72 during chemical etching. Further MERL 711 matrix produced the best VHP compacts and TLP bonding at all temperatures while maintaining SiC grit integrity.

Blade Tip Coating Properties

The selected M-CrAlY matrix materials exhibited a range of corrosion/oxidation resistance and varying degrees of strength. In general, the higher-strength at temperature materials possess somewhat less corrosion resistance than the lower-strength materials.

A general ranking of candidate tip alloy materials (MERL 711, MERL 72, and TIPALLOY I) as determined from tests in cyclic hot corrosion and oxidation is given in table 3.

Cyclic hot corrosion testing (20-hr cycles) was performed at 1835°F for a total of 100 hr. A supersaturated water solution of Na_2SO_4 in the amount of 1 mg/cm² was applied to the specimen surface before each 20-hr cycle. Between cycles the specimens were cooled, washed, and weighed to measure any weight changes. The tested specimens were examined metallographically to characterize their corrosion behavior.

Table 2. Single-Bladed Rub Evaluation Summary

Grit (Unetched) (%)	Volume Wear Factor (Before Transfer)	Maximum Seal Wear (Mils)	Interference (Mils)	Matrix Material
30	0.13	2.4	3.9	MERL 711
30	0.13	2.3	3.8	MERL 72
40	0.18	1.8	3.3	MERL 711
40	0.17	1.9	3.4	MERL 72
50	0.11	2.8	4.3	MERL 711
50	0.14	2.2	3.7	MERL 72
Grit (Etched) (%)				
50	0.085	4.5	9.5	MERL 711
50	0.055	5.5	10.0	MERL 72

Table 3. Summary of Alloy Performance in Cyclic Hot Corrosion at 1835°F and Cyclic Oxidation at 2000°F and at 2100°F

Test Conditions	Group I	Group II	Group III
	Exhibits Minimal or No Internal Corrosion/Oxidation and/or Minimal or No Oxide Spallation	Exhibits Some Internal Corrosion/Oxidation and/or Some Oxide Spallation	Exhibits Massive Internal Corrosion/Oxidation and/or Massive Oxide Spallation
100 hr Cyclic (20 hr) Hot Corrosion Test at 1835°F		TIPALLOY I (VHP) MERL 711 (cast or VHP) TIPALLOY I (cast) MERL 72 (cast or VHP)	PWA 1422
100 hr Cyclic (20 hr) Oxidation Test at 2000°F	MERL 711 MERL 72	PWA 1422	
100 Hr cyclic (20 hr) Oxidation Test at 2100°F	MERL 711 (VHP) TIPALLOY I (VHP or cast)	MERL 72 (VHP) PWA 1422 (cast)	

Cyclic oxidation testing was conducted at 2000 and 2100°F. The specimens were cooled and weighed after each 20-hr cycle until 100 hr were completed. The tested specimens were examined metallographically to characterize oxidation behavior.

The oxidation and hot corrosion resistance of vacuum hot pressed (VHP) MERL 711 and VHP TIPALLOY I are comparable to their cast counterparts except that VHP materials are not as susceptible to intergranular attack under similar test conditions. The performance of TIPALLOY I was comparable to MERL 711. The corrosion resistance and susceptibility of VHP MERL 72 was less than that of cast MERL 72 in hot corrosion testing at 1835°F. The VHP MERL 72 specimen oxidation tested at 2100°F showed heavy general oxidation attack.

Hot hardness measurements were analyzed for the three alloys to indicate their relative high-temperature strength. The hot hardness testing was performed at room temperature, 500, 1000, 1200, 1400, 1600, 1800, and 2000°F in a vacuum. The hardness data reflected the range of material strength available in these alloy systems. The results of this evaluation are illustrated in figure 29.

Physical properties were measured for the SiC grit/MERL 711 compact system by utilizing per cent concentration estimates from each constituent. The density (ρ) for the 50 vol% SiC abrasive grit/MERL 711 matrix compact is approximately 0.217 lb/in.³. Thermal conductivity (k) and thermal expansion (α) curves that vary with percent abrasive grit concentration are depicted in figures 30 and 31.

Stress Rupture Evaluation

Six comparison stress rupture compacts were TLP bonded to PWA 1422 bars in the same bond run as the 12 rig blades. Three MERL 711 specimens (no abrasive grits) and the three 50 vol% Al₂O₃ sputtered SiC (8-mil diameter) grits in MERL 711 were heat-treated to simulate engine turbine hardware preparation. Visual inspection before machining and fluorescent penetrant inspection after machining indicated minor bond defects but were considered acceptable except for a compact containing SiC grits which failed at the TLP bond during the preparation phase of the stress rupture testing.

Results of the stress-rupture testing for compacts of MERL 711 and 50 vol% Al₂O₃ sputtered SiC in MERL 711 are given in table 4. Room temperature (RT) ultimate tensile strength of the 50 vol% Al₂O₃ sputtered SiC grits in MERL 711 was 16.8 ksi which is approximately 11% of the ultimate tensile strength of solid MERL 711 (146.7 ksi). The stress rupture life for the 50 vol% Al₂O₃ sputtered SiC grit in MERL 711 was considerably lower than that of the solid MERL 711. This reduction can be attributed to the relatively high volume percent of abrasive grits in the compact.

Figure 32 illustrates a microsection of the failed 1800°F MERL 711 specimen which ruptured after 11.4 hr. Secondary cracking is prevalent near the plane of rupture. Figure 33 illustrates the rupture area of the 50 vol% Al₂O₃ sputtered SiC grits in the MERL 711 stress rupture sample which failed after 0.9 hr at 1800°F and 2 ksi.

A comparison of this 50 vol% 8-mil SiC grit (MERL 711) limited tensile/stress rupture with limited data for 15- and 25-mil Al₂O₃ grits in MERL 711 matrix (unpublished) extrapolates to a predicted life of over 100 hr for 1 ksi stress at an 1800°F blade temperature. It is obvious that additional tensile/stress rupture data is required. In addition, reduced volume percent SiC grits may be necessary to increase stress rupture life which in turn would require additional rub test data to determine abrasive tip wear effectiveness.

Table 4. Stress Rupture/Tensile Test Results

Tensile

<i>Specimen Type</i>	<i>Test Temperature (°F)</i>	<i>Ultimate Tensile Strength (ksi)</i>	<i>Rupture Zone</i>
MERL 711	RT	146.7	MERL 711
MERL 711	1800	24.2	Bond
50 vol% SiC-MERL 711	RT	16.8	Grits

Stress Rupture

<i>Specimen Type</i>	<i>Test Temperature (°F)</i>	<i>Hours to Failure</i>	<i>Rupture Zone</i>
MERL 711	1800	11.4	MERL 711
MERL 711	1800	12.8	MERL 711
50 vol% SiC-MERL 711	1800	0.9	Grits

Thermal Shock - Full-Scale Blades

Two blade tips of 50 vol% Al_2O_3 sputtered SiC in MERL 711 were vacuum hot pressed at 2100°F and 5 ksi for 1 hr. The compacts were machined prior to TLP bonding to the prepared tips of the JT9D (PWA 1422) first-stage geometry turbine blades. The blade tips were then TLP bonded to PWA 1422 turbine engine blades. The resulting bonds were unsatisfactory and exhibited large gaps in the bonds on both sides of the airfoil, particularly at the leading and trailing edges. Scale-up to engine hardware from rig hardware with the 50 vol% SiC grit abrasive produced inadequate positive loading of the leading and trailing edges of the engine blade. Fixturing adjustments were required to produce adequate positive loading of the abrasive tip compact on the engine blade during the TLP cycle. The abrasive tips were remachined and TLP bonded with proper fixturing to new PWA 1422 test blades. The use of a molybdenum cap to apply a uniform positive loading to the blade tips during the bond cycle produced acceptable bonds. The blades were airfoil and tip coated with a protective coating before initiating fluidized-bed thermal fatigue testing. An overall view of the PWA 1422 blade with the tip coating is presented in figure 34. Additional views of the blade tip (figures 35 and 36) illustrate the high concentration and good distribution of abrasive grits in the compact.

The thermal shock test was conducted in a fluidized bed with both the abrasive blade tips being cycled from essentially 100°F to 1800°F simultaneously to a goal of 500 cycles. At the 463 cycle point, the thermal shock tester malfunctioned resulting in an interruption of the cyclic evaluation. Photographs were taken of the tested blades at that point. One blade sustained a solid impact against the fluidized bed support when the tester arm malfunctioned. This resulted in a chip in the trailing edge of the abrasive tip treatment, as shown in figure 37. The second blade escaped the impact and did not exhibit any observable problem areas (figure 38). Post-test Zygo results revealed no apparent bond or compact problems for either blade except for the chipped location on one of the blade tips. Thermal shock tests were resumed after repair of the rig thus completing the 500 cycle test.

This fluidized bed test provided initial verification that the selected abrasive blade tip treatment coating can withstand numerous thermal shock cycles with minimal adverse effects to compact or bond integrity.

PHASE II - ENGINE SIMULATION EVALUATION OF BLADE TIP COATING

Material and Process Combination

The best material and process combination that evolved from the Phase I effort was the 50 vol% SiC grits with a 1600°F sputtered Al_2O_3 coating in a MERL 711 matrix. The 1600°F sputtered Al_2O_3 coating on SiC grits produced a 100% effective diffusion barrier in MERL 711 and was 90% effective through the full TLP bond and heat-treat cycle currently required for high-strength bonds (2 hr at 2200°F). The other diffusion barrier materials/processes and matrix alloys were shown to be inferior. Chemically etched 50 vol% SiC grits produced the best wear effectiveness in single-blade rub tests.

Multi-Bladed Dynamic Rub Tests

Multi-bladed tests allow for improved simulation of engine rub interactions in terms of blade passing frequency (BPF) which allows duplication of the amount of material removed from the seal per blade pass. The multi-bladed tests conducted in the rig shown in figure 27 were performed with the ceramic turbine seal developed in NAPTIC Contract N00140-74-C-0586.

Twelve rig blade tips of 50 vol% Al_2O_3 , sputtered SiC, 8-mil diameter grits in MERL 711 metal matrices were vacuum hot pressed at 2100°F and 5 ksi for 1 hr. Figure 39 illustrates a typical dispersion of Al_2O_3 , sputtered SiC grits at 50 vol% in the MERL 711 matrix. Minimal reaction was observed between the SiC grits and the MERL 711. The tips were machined, pre-oxidized for 1 hr at 2100°F and TLP bonded to PWA 1422 rub rig blades. All rig blades were contoured to remove excess tip material and fluorescent penetrant inspected for bond integrity. Approximately 3 to 4 mils of matrix material was chemically etched from the blade tip surface, thereby exposing only abrasive grits at that location.

The first multi-blade test was conducted at room temperature, a surface speed of 1000 ft/sec and an interference rate of 0.001 in./sec. The average measured seal wear was recorded at 0.0165 in. (figure 40). The average blade wear for the six blades was 0.002 in. The tested blade tips remained intact with no visible indications of compact or bond instabilities. The effectiveness of the multi-bladed etched system can be measured in two areas. First, the blade tip did not begin depositing matrix material on the seal until an interference of 0.0185 in. had been achieved. This is twice the effective interference over single-bladed etched evaluations with less relative clearance increase. Secondly, the rate of the rub-induced time temperature effects of the blade tip were considerably lower with the additional blades rubbing. This effect increased the length of interaction time before incipient tip transfer was initiated.

The second multi-blade test was conducted at a steady-state seal temperature of approximately 2800°F. The average measured seal wear was 0.0135 in. and average blade wear was recorded at 0.0025 in. (figure 41). The blade tip temperature at steady-state before interaction was somewhat less than 1200°F. Post-test appearance of the blade tips revealed that the abrasive grits were worn but still intact. One blade began transferring to the seal after wearing away its exposed grits but the compact and bond remained structurally sound. Figure 42 illustrates the increased ceramic turbine seal wear from unetched single bladed and etched multi-bladed tests with essentially constant blade wear. Therefore, SiC grits have constant wear effectiveness until the metal matrix becomes active in the rub. Furthermore, figure 43 illustrates the effectiveness of free SiC grits in producing increased ceramic wear (increased interference) and the rapid detrimental aspects of the metal matrix associated with the rapid temperature excursion of the blade tip.

The selected tip treatment system, 50 vol% SiC/MERL 711 compact, exhibited extremely favorable rub results in conjunction with the Navy-sponsored ceramic seal material. Minimal engine clearance penalties would be realized for local blade/seal interferences up to 20 to 30 mils.

SECTION III CONCLUSIONS

The abrasive blade tip treatment concept to provide blade tip wear resistance for interferences with advanced turbine ceramic outer air seals has been proven feasible. The blade tip treatment was successful in wearing the ceramic seals with minimal blade tip wear and no blade transfer. The dynamic impact structural integrity of the compacts was upheld throughout all rub evaluations.

The 1600°F RF sputter coated Al_2O_3 on the SiC abrasive grits was the only effective protective barrier coating. This (approximately 0.1 mil) coating virtually eliminated SiC grit reaction with the surrounding metal matrix and subsequent grit dissolution. The CVD process using HfO_2 and Al_2O_3 resulted in very limited SiC coating success. Grit coating was sparse and provided locations for grit reaction and dissolution.

The MERL 711 alloy material was found to be the most effective matrix for all possible considerations. This matrix material withstood the VHP fabrication, TLP bond and heat treatment (2 hr at 2200°F) cycles with minimal degradation to the total compact system. MERL 711 ranked better or at least comparable with the other candidate tip alloy materials in cyclic hot corrosion and cyclic oxidation testing. It also revealed better structural integrity than the MERL 72 candidate when subjected to a tip surface chemical etching process. The TIPALLOY I matrix material exhibited inability to withstand the minimum TLP bond cycle without promoting SiC grit dissolution. The selected blade tip coating configuration is illustrated in figure 44.

Full-scale blade TLP bonding required revised fixturing to assure positive loading for adequate quality bonds. The preliminary scale-up attempt produced inadequate positive loading of the leading and trailing edges of advanced engine blades (curling). The use of a molybdenum cap to apply a uniform positive loading to the engine configuration blade tips during the bond cycle produced acceptable bonds.

Fluidized bed/thermal shock tests on the 50 vol% abrasive SiC grit/MERL 711 compact system provided preliminary verification that the abrasive tip treatment could withstand numerous thermal shock cycles with minimal adverse effects to the compact or bond integrity.

Stress rupture life for the selected abrasive blade tip treatment system may be marginal. A preliminary predicted engine life of over 100 hr for 1.0 ksi at 1800°F blade temperature has been estimated.

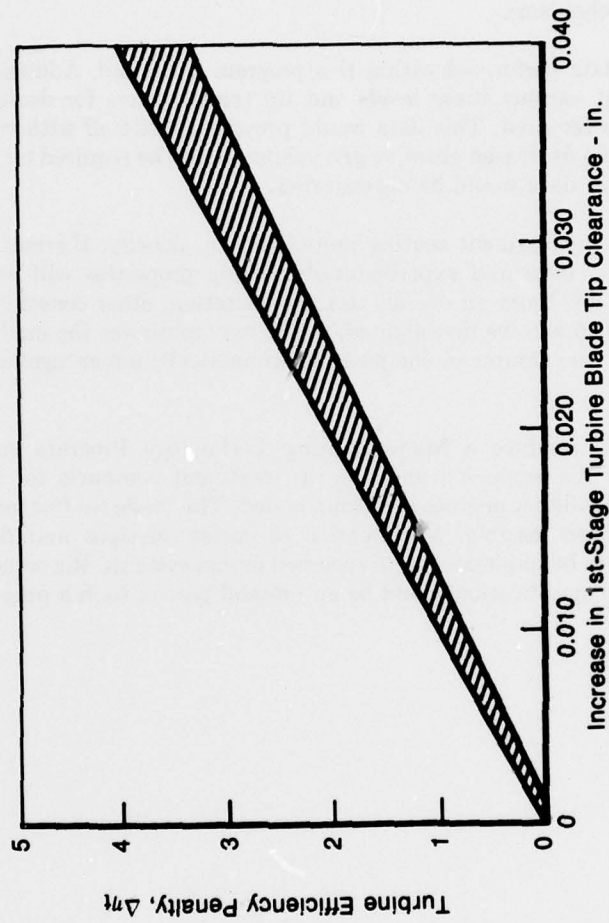
SECTION IV RECOMMENDATIONS

A preliminary attempt to provide abrasive tip treatment/ceramic seal wear technology has been accomplished in this contract. Additional effort is still required to accurately define rig to engine blade/seal wear scale-up parameters. Engine blade pass frequency/chip thickness, steady-state blade tip temperatures and other dynamic parameters must be simulated and evaluated to determine critical areas which define the blade/seal system wear characteristics. An accurate wear prediction system is required for each engine design system in order to define clearances and interferences allowable to accommodate maximum seal wear with low blade tip wear and no blade tip transfer mechanisms.

Stress rupture data performed within this program is limited. Additional information to define compact life at various stress levels and tip temperatures for decreased abrasive grit volume percentages is required. This data would provide a trade-off with rupture life and grit volume percentages. If a decreased abrasive grit volume would be required for a particular design system, blade/seal wear data would be necessitated.

Abrasive blade tip treatment coating properties, i.e. density, thermal expansion, tensile strength, etc. must be measured experimentally. These properties will be required for any particular design system. From an overall cost consideration, other consolidation and bonding techniques should continue to be investigated, which may minimize the intricate and expensive fabrication processes. An example of one possible economically advantageous joining technique is diffusion bonding.

Consideration to conduct a Manufacturing Technology Program to develop a mini-production capability for producing abrasive tip treatment compacts for utilization in both current and advanced military engines is recommended. The blade tip treatment coating system process has been proven feasible. Modification of current designs and developing scale-up hardware would have to be implemented to specified design systems. Rig component evaluations and material property specification would be an integral part of such a program.



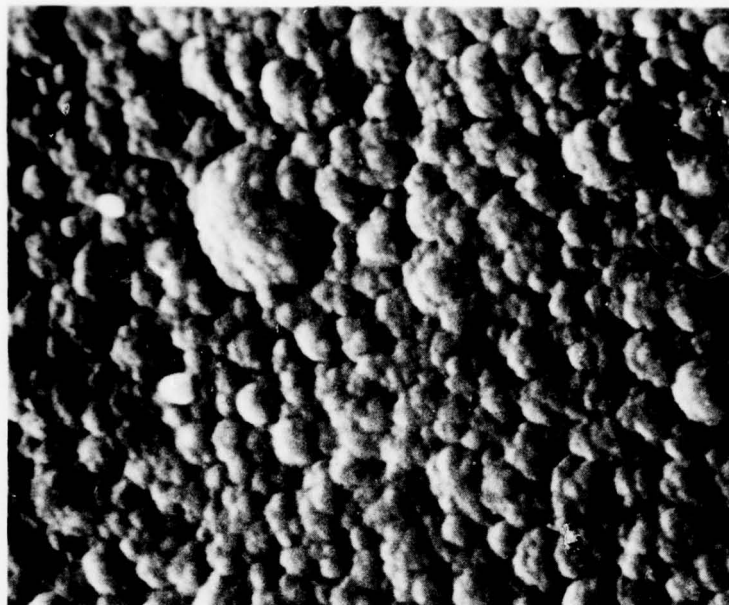
FD 111010

Figure 1. Turbine Efficiency Penalty vs Blade Tip Clearance



Magnification : 300X

A

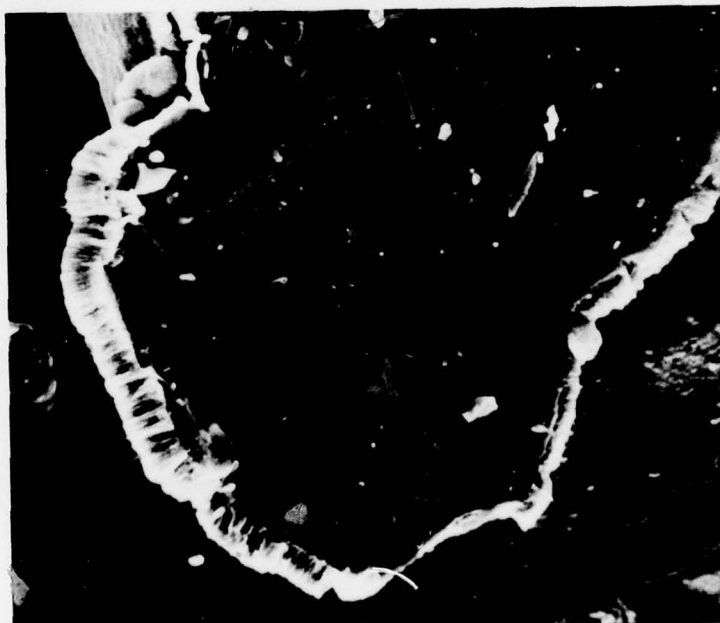


Magnification : 10,000X

B

FD 111011

Figure 2. Alumina Coated Silicon Carbide Grains As-Coated From the RF Sputtering Run at 1200°F, 600w, 2-in. Dia. Target for 95 Hr



Magnification: 1000X A



Magnification: 3000X B

FD 111012

Figure 3. Alumina Coated Silicon Carbide Grains Mechanically Fractured From the RF Sputtering Run at 1200°F, 600w, 2-in. Dia. Target for 95 Hr



Magnification : 100X

A

Magnification : 500X

B

FD 111013

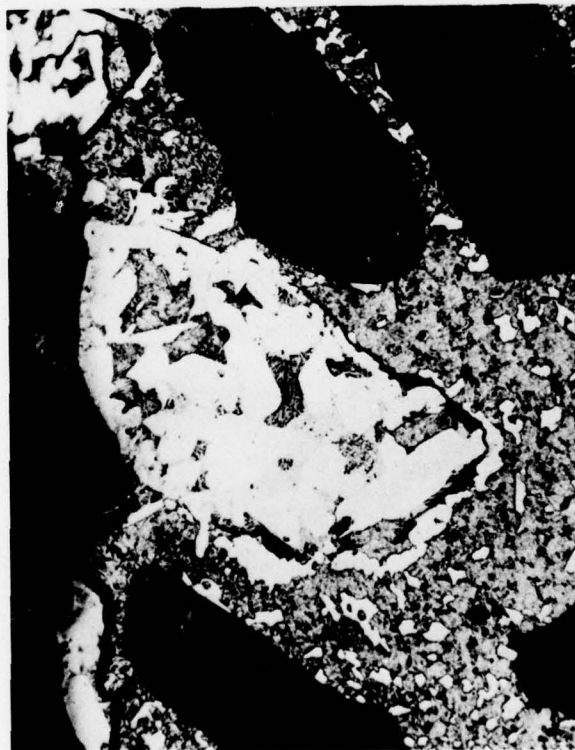
Figure 4. Alumina Coated Silicon Carbide Grains From the RF Sputtering Run As-Vacuum Hot Pressed at 2100°F, 1 Hr, 5 ksi in MERL 711 Co Base Alloy



Magnification: 25X

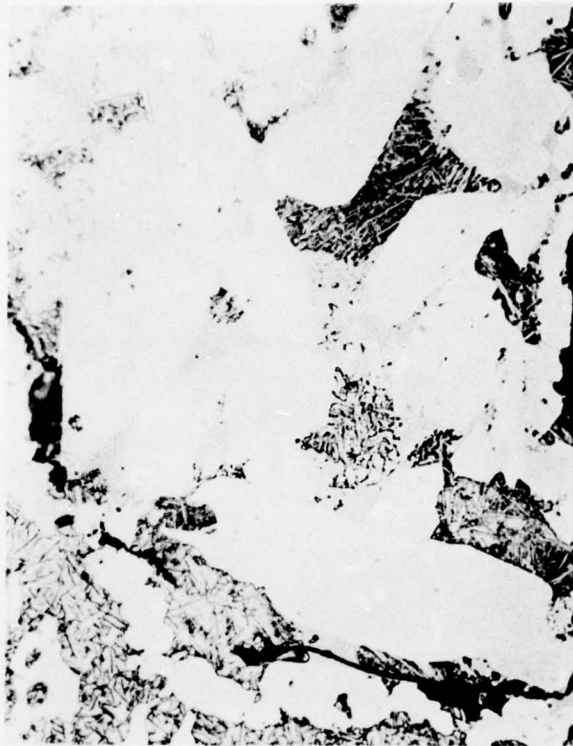
FD 111014

Figure 5. Thirty Percent by Volume Al_2O_3 Sputtered SiC at 1200°F in MERL 711 After TLP® Bonding to PWA 1422 Blade and Solution Heat Treatment



Magnification: 200X

A



Magnification: 500X

B

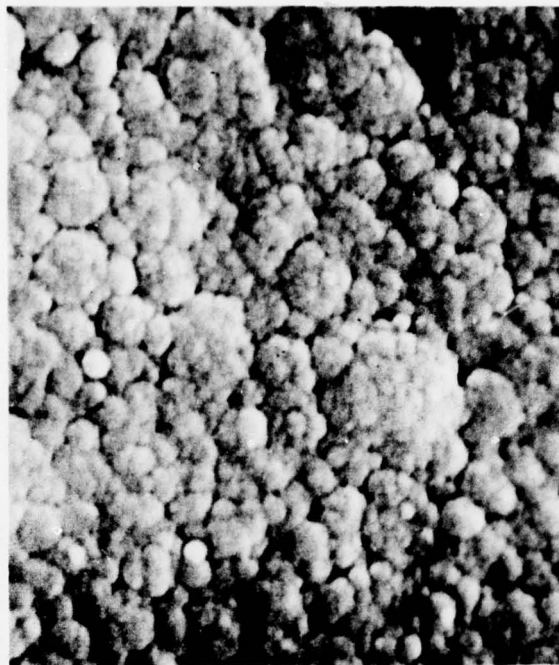
FD 111015

Figure 6. Thirty Percent by Volume Al_2O_3 Sputtered SiC at 1200°F in MERL 711 After TLP® Bonding to PWA 1422 Blade and Solution Heat Treatment



Magnification: 300X

A

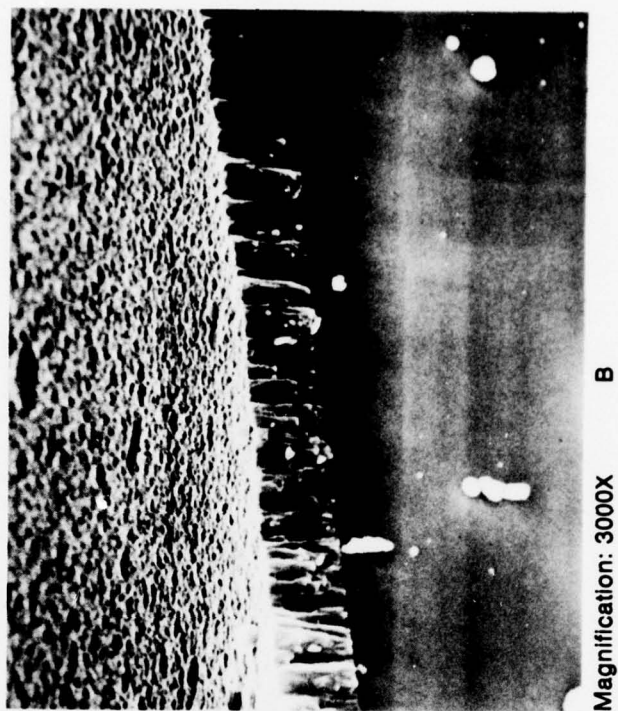


Magnification: 10,000X

B

FD 111016

Figure 7. Alumina Coated SiC Grits From the RF Sputtering Run at 1600°F, 600w, 2-in. Dia. Target for 94 Hr

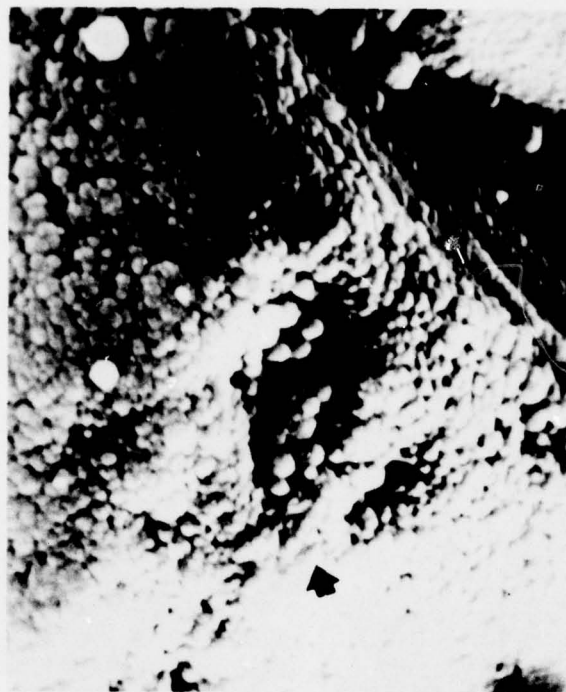


FD 111017

Figure 8. Alumina Coated SiC Grits From the 1600°F Sputtering Run Fractured to Show the Coating Cross Section



Magnification: 1000X A



Magnification: 3000X B

FD 111018

Figure 9. Alumina Coated SiC Grits From the 1600°F Sputtering Run Showing a Geometry-Related Coating Defect



Magnification: 100X

A



Magnification: 500X

B

FD 111019

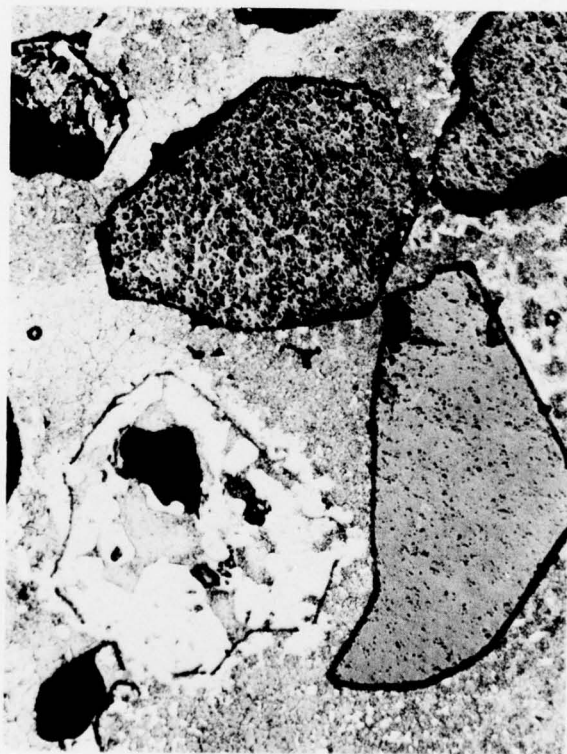
Figure 10. Alumina Coated SiC Grits From the 1600°F RF Sputtering Run as Vacuum Hot Pressed at 2100°F, 1 Hr, 5 ksi in MERL 711 Co Base Alloy



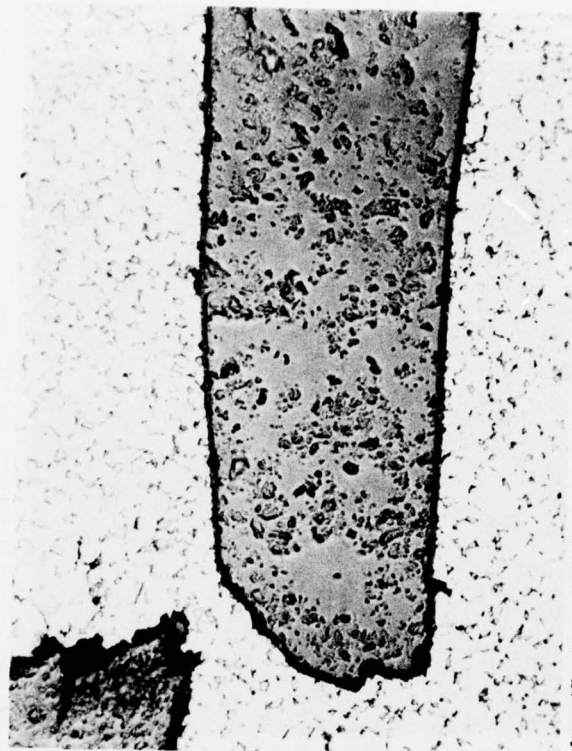
Magnification: 25X

FD 111020

Figure 11. Thirty Percent by Volume Al_2O_3 Sputtered SiC at 1600°F in MERL 711 After TLP[®] Bonding to PWA 1422 Blade and Solution Heat Treatment



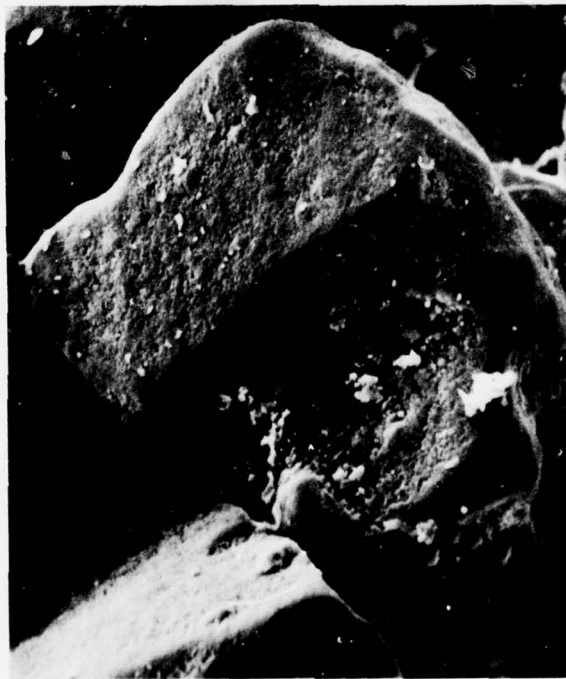
A



B

FD 111021

Figure 12. Thirty Percent by Volume Al_2O_3 Sputtered SiC at 1600°F in MERL 711 After TLP® Bonding to PWA 1422 Blade and Solution Heat Treatment



Magnification: 300X

A



Magnification: 10,000X

B

FD 111022

Figure 13. *HfC+ C Coated SiC Grits — Chemical Vapor Deposition Coating*



Magnification: 1000X A



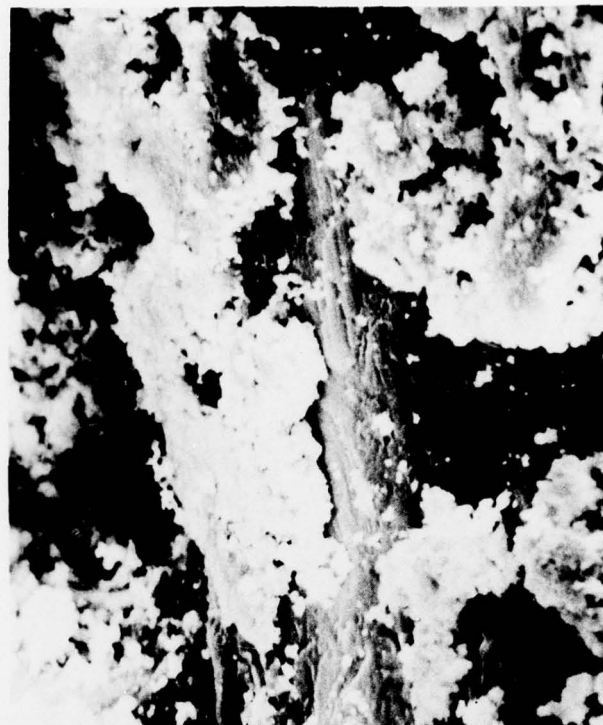
Magnification: 3000X B

FD 111023

Figure 14. HfC+C Coated SiC Grits — Chemical Vapor Deposition Coating Fractured to Show the Coating Cross Section



Magnification: 300X



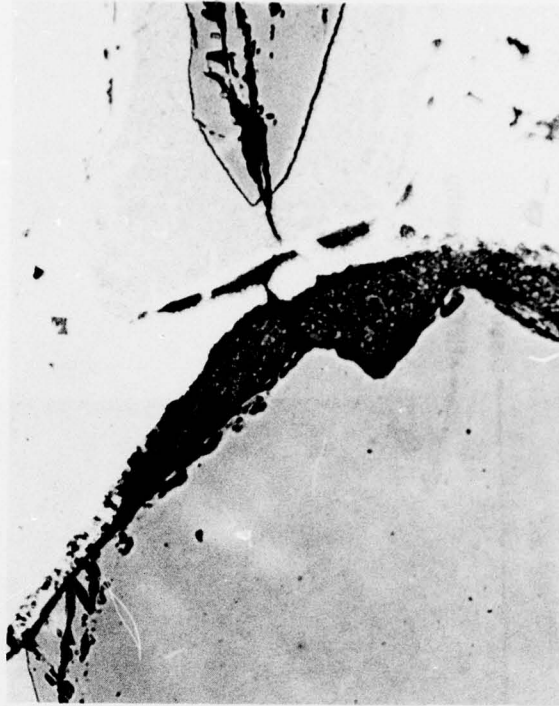
Magnification: 1000X

FD 111024

Figure 15. Scanning Electron Microscope Image of Oxidized $\text{HfC}+\text{C}$ Coated SiC Showing Spotty HfO_2 on SiC



Magnification: 100X A



Magnification: 500X B

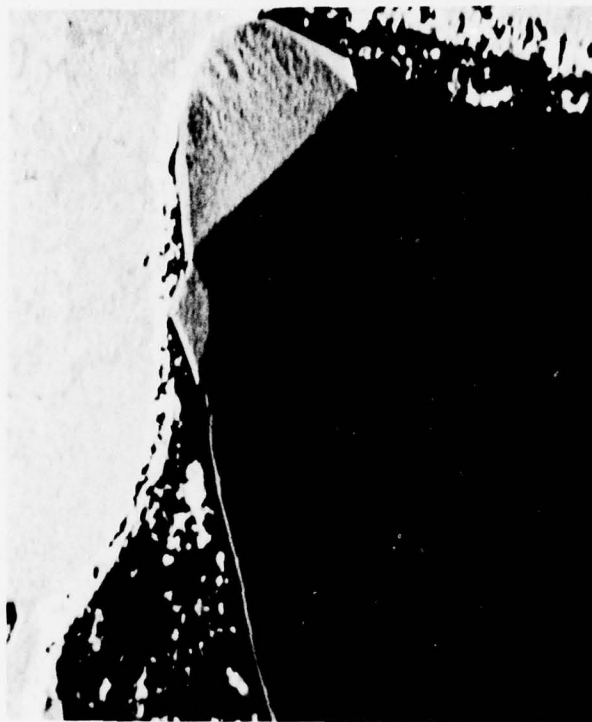
FD 111025

Figure 16. H/C+C Coated SiC Grits as Vacuum Hot Pressed at 2100°F, 1 Hr, 5 ksi in MERL 711 Co Base Alloy



FD 111026

Figure 17. Thirty Percent by Volume HfC+C Coated SiC and MERL 711 Vacuum Hot Pressed at 2100°F, 1 Hr, 5 ksi



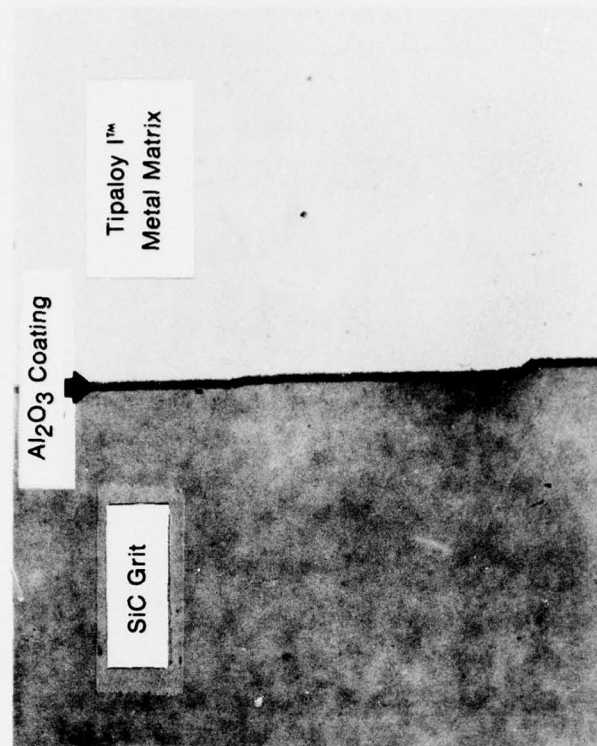
Magnification: 800X

FD 111027

Figure 18. Inverted Image of Area in Figure 17 as Viewed in Scanning Electron Microscope Showing SiC Grit With Reacted and Unreacted HfC+C Coating in MERL 711



Magnification: 100X



Magnification: 500X

FD 111028

Figure 19. Thirty Volume Percent Al_2O_3 Coated SiC Grits Vacuum Hot Pressed in TIPALOY I™

Tipaloy I™ Metal
Matrix With
Porosity Evident

Al₂O₃
Coating

SiC Grit

Magnification: 500X

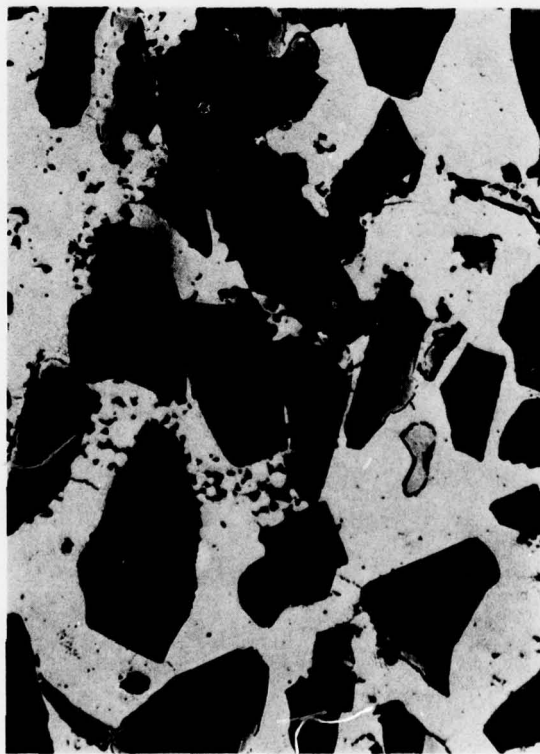
FD 111029



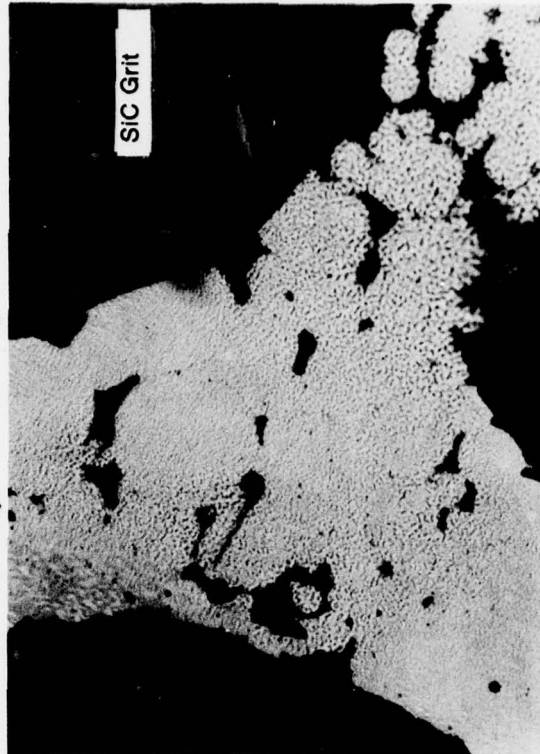
Magnification: 100X

Figure 20. Forty Volume Percent Al₂O₃ Coated SiC Grits Vacuum Hot Pressed in TIPALLOY I™

Tipaloy I[™] Metal
Matrix With
Porosity Evident Al₂O₃ Coating



Magnification: 100X



Magnification: 500X

FD 111030

Figure 21. Fifty Volume Percent Al₂O₃ Coated SiC Grits Vacuum Hot Pressed in TIPALOY I[™]



Magnification: 100X



Magnification: 500X

FD 111031

Figure 22. Thirty Volume Percent Al_2O_3 Coated SiC Grits Vacuum Hot Pressed in MERL 72



Magnification: 100X



Magnification: 500X

FD 111082

Figure 23. Forty Volume Percent Al_2O_3 Coated SiC Grits Vacuum Hot Pressed in MERL 72



Magnification: 100X



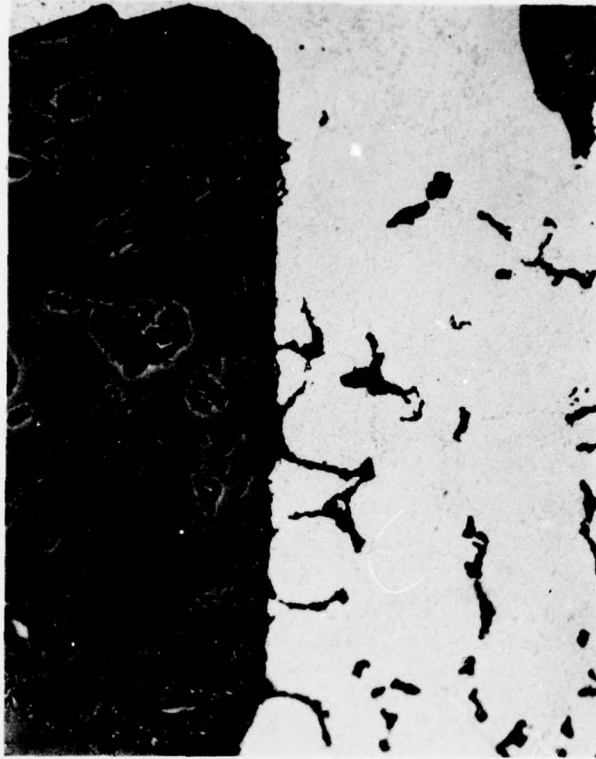
Magnification: 500X

FD 111033

Figure 24. Fifty Volume Percent Al_2O_3 Coated SiC Grits Vacuum Hot Pressed in MERL 72



Magnification: 100X



Magnification: 500X

FD 111034

Figure 25. Forty Volume Percent Al_2O_3 Sputtered SiC in MERL 72 TLP₈ Bonded at 2000°F, 8 Hr Showing Minimal Reaction Between the SiC and MERL 72



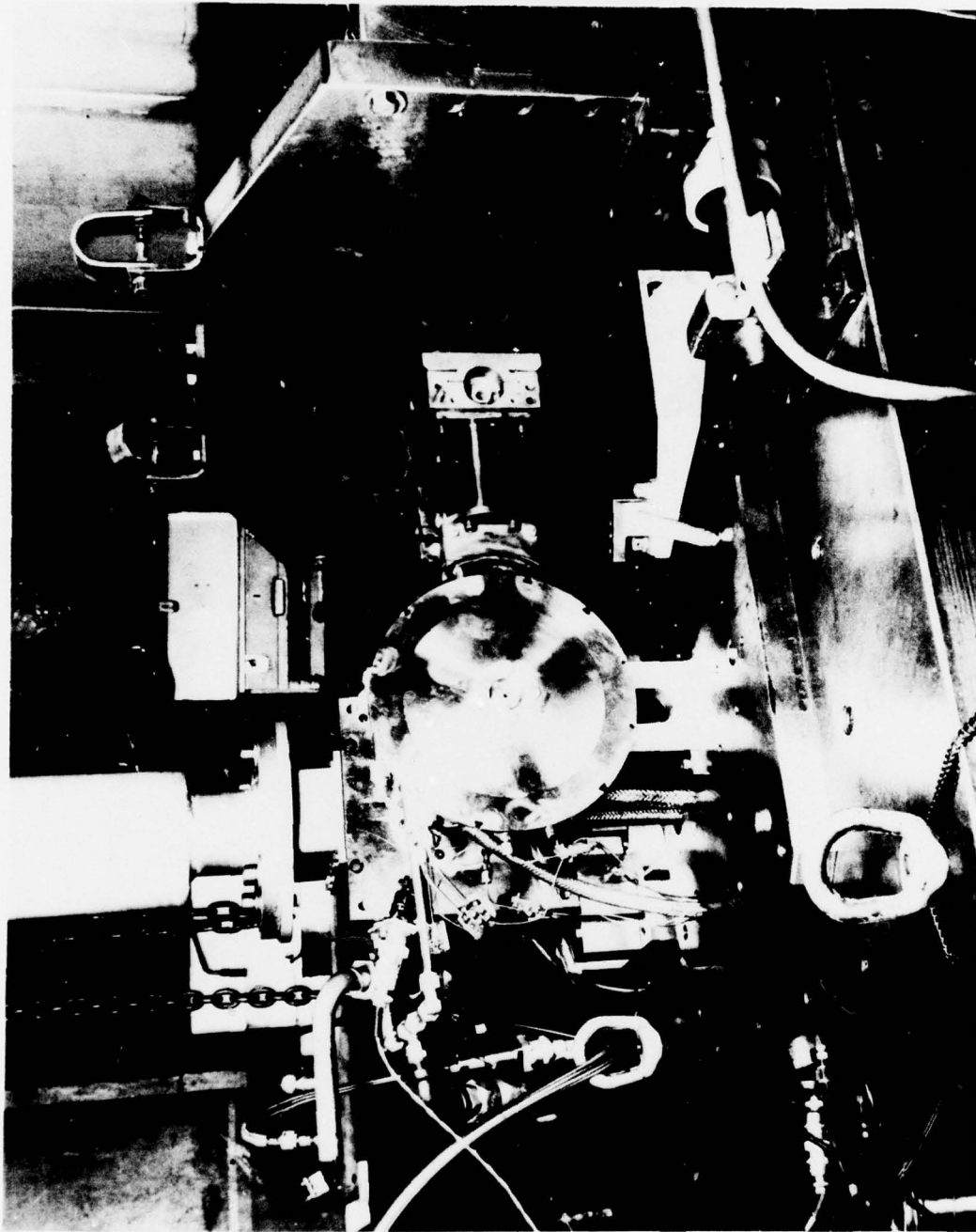
Magnification: 100X



Magnification: 500X

FD 111035

Figure 26. Forty Volume Percent Al_2O_3 Sputtered SiC in TIPALLOY ITM TLP[®] Bonded at 2000°F, 8 Hr Showing Reaction Between the SiC and TIPALLOY I



FC 35372A

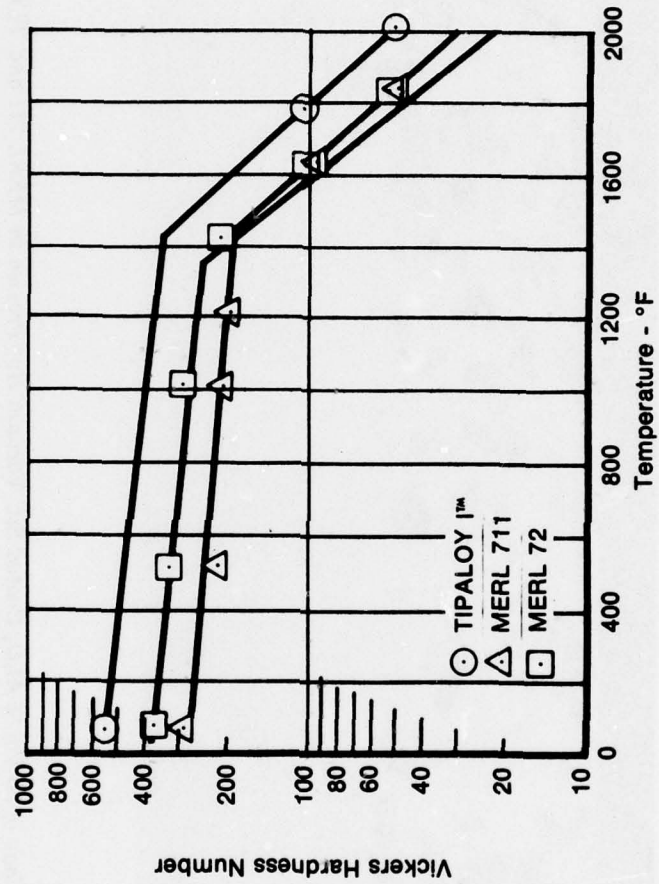
Figure 27. Dynamic Abradability Rub Rig



Magnification: 25X

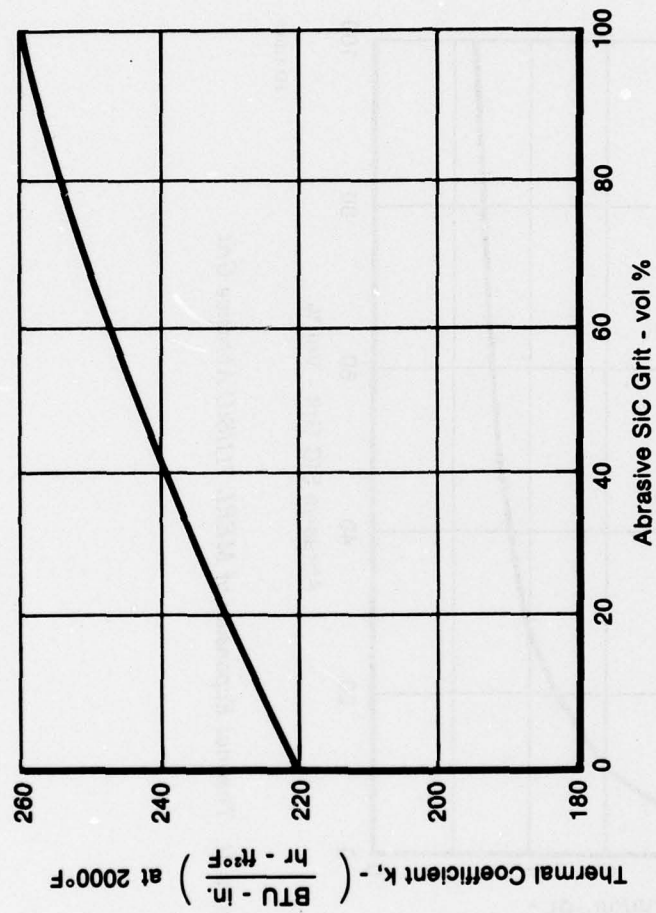
FD 111036

Figure 28. Rub Tested Rig Blade of 30 Vol % Al_2O_3 Coated SiC Vacuum Hot Pressed in MERL 711 and TLP[®] Bonded



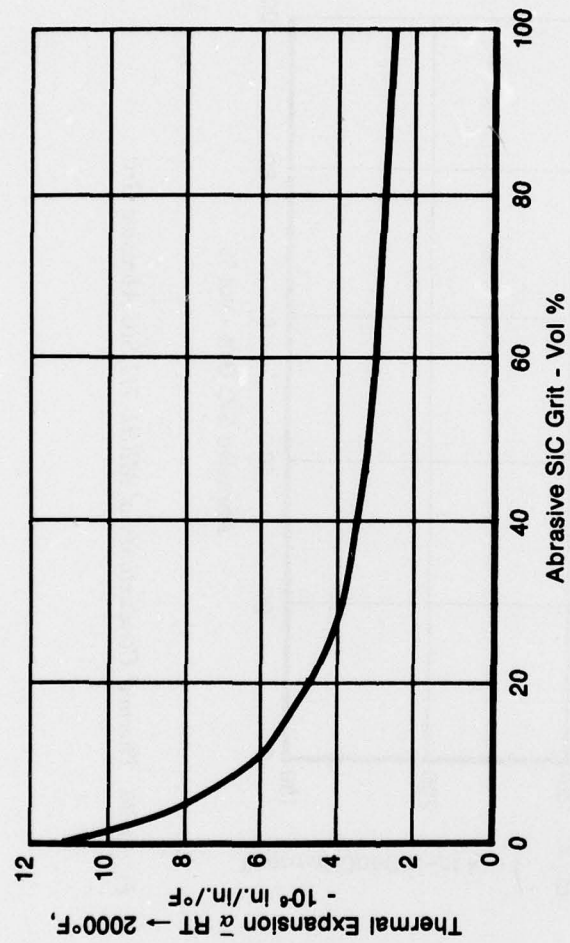
FD 111087

Figure 29. Hot Hardness of Selected M-CrAlY Alloys



FD 111068

Figure 30. Thermal Conductivity of MERL 711/SiC Abrasive Grit

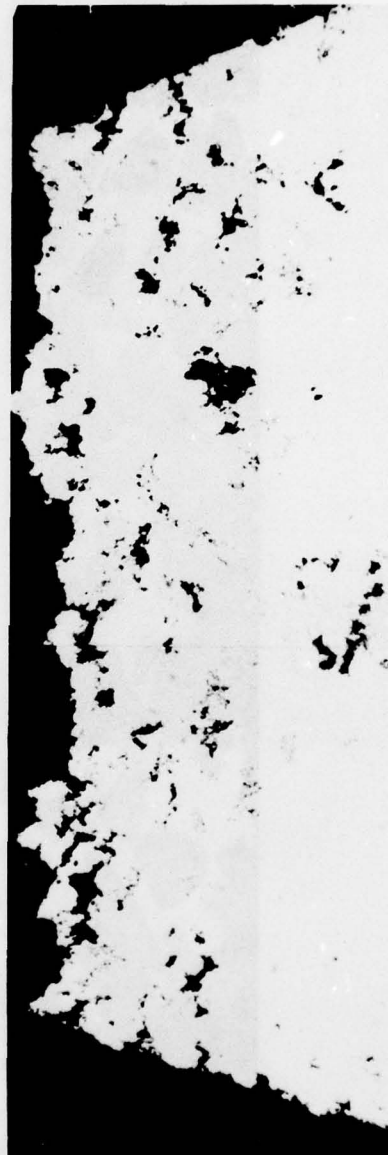


FD 111038

Figure 31. Thermal Expansion of MERL 711/SiC Abrasive Grit



Magnification: 50X



Magnification: 100X

FD 111040

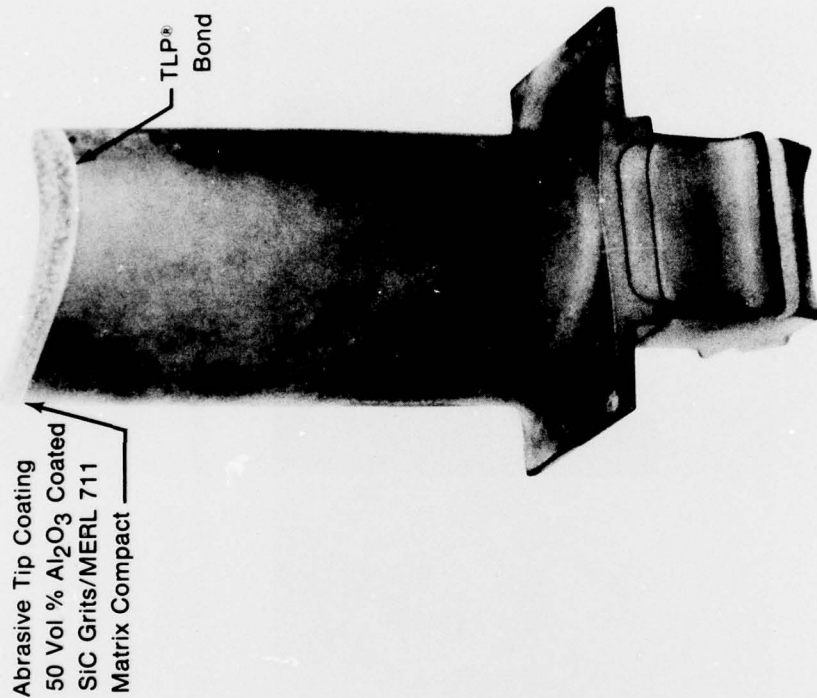
Figure 32. Stress Rupture of MERL 711 at 1800°F, 11.4 Hr



Magnification: 50X

FD 111041

Figure 33. Stress Rupture of 50 Vol % SiC Grits/MERL 711 Matrix at 1800°F, 0.9 Hr



Abrasive Tip Coating
50 Vol % Al_2O_3 Coated
SiC Grits/MERL 711
Matrix Compact

TLP®
Bond

FD 111042

Figure 34. Engine-Size Blade (PWA 1422) Used for Thermal Fatigue Evaluation



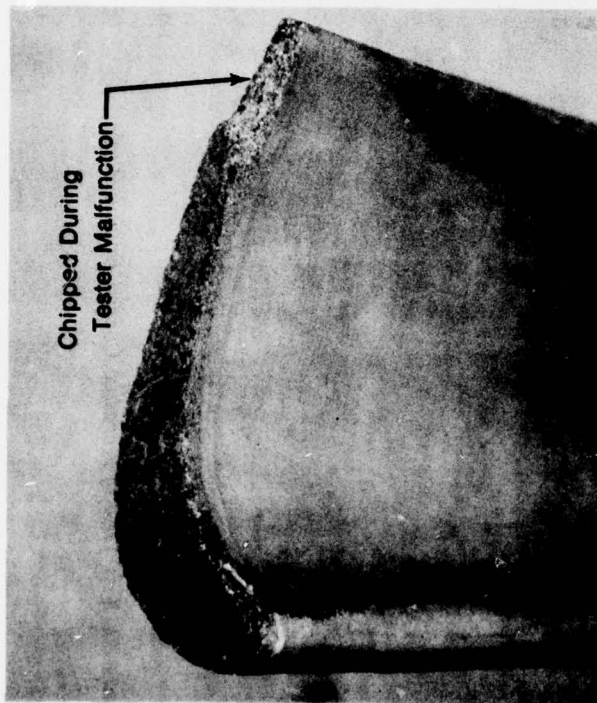
FAL 40846A

Figure 35. Oblique View Showing Abrasive Coating and Bond on Engine Blade



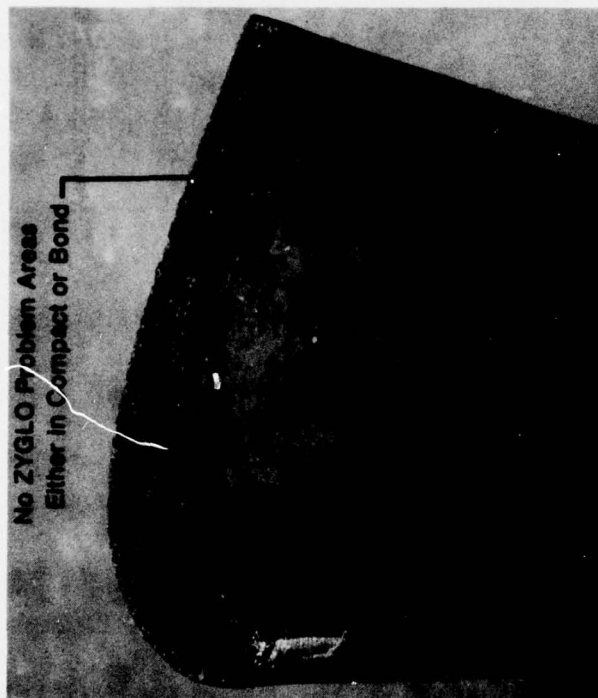
FAL 40845A

Figure 36. Top View of Abrasive Coated Engine Blade



FD 111043

Figure 37. Engine Blade Compact After 463 Thermal Shock Cycles



FD 111044

Figure 38. Engine Blade Compact After 463 Thermal Shock Cycles



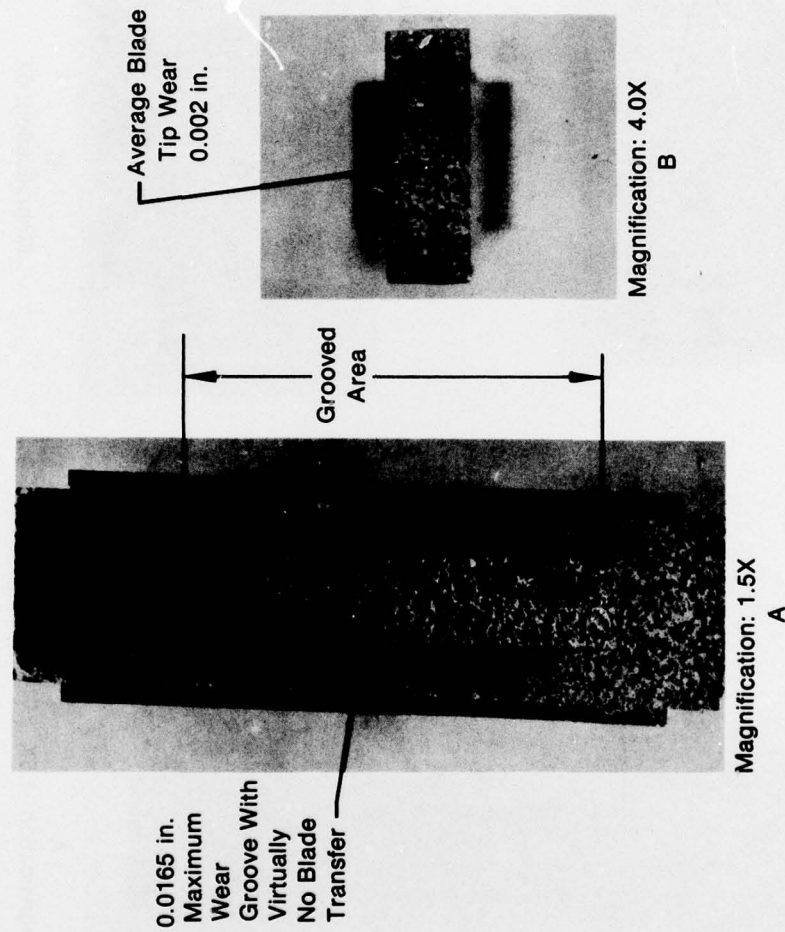
Magnification: 50X



Magnification: 200X

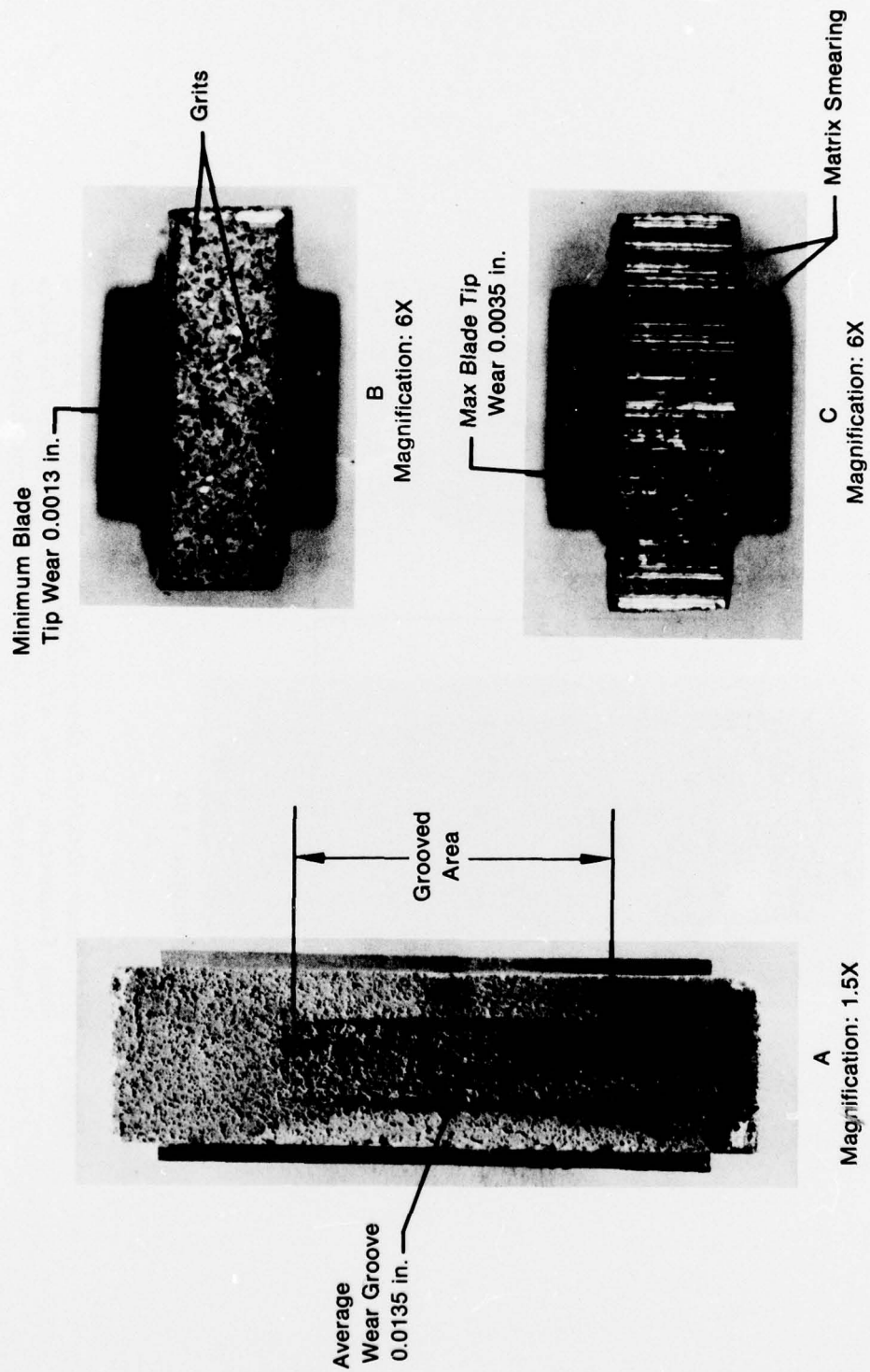
FD 111045

Figure 39. Fifty Volume Percent Al₂O₃ Sputtered SiC in MERL 711 Matrix Showing Good Al₂O₃ Coating and No Grit Reaction



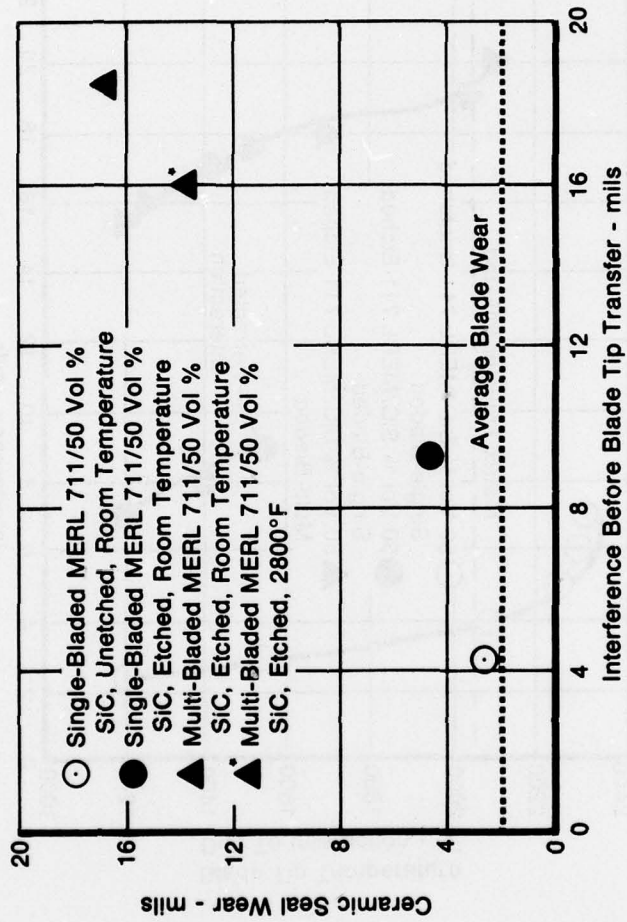
FD 111046

Figure 40. (A) Ceramic ($ZrO_2/NiCr$ Graded Structure) Rig Segment Rubbed at Room Temperature by Six SiC/Al_2O_3 , MERL 711 Coated Rig Blades (Chemically Etched), and (B) Corresponding (Typical) Rubbed Blade With Grits Intact and Minimal Matrix Smearing



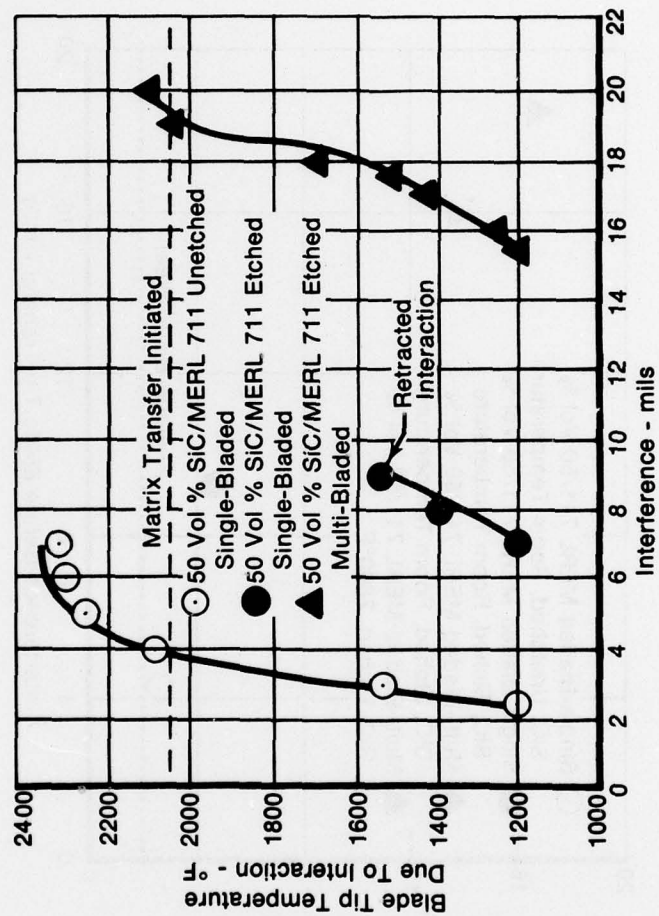
FD 111047

Figure 41. (A) Ceramic Segment Tested at Maximum Observed Temperature of 2800°F Rubbed by Six SiC/Al₂O₃ MERL 711 Coated Rig Blades (Chemically Etched), (B) Tested Blade Exhibiting Minimum Tip Wear Revealing Grits are Unaffected by Rub, and (C) Tested Blade Tip Exhibiting Maximum Tip Wear and Incipient Matrix Smearing



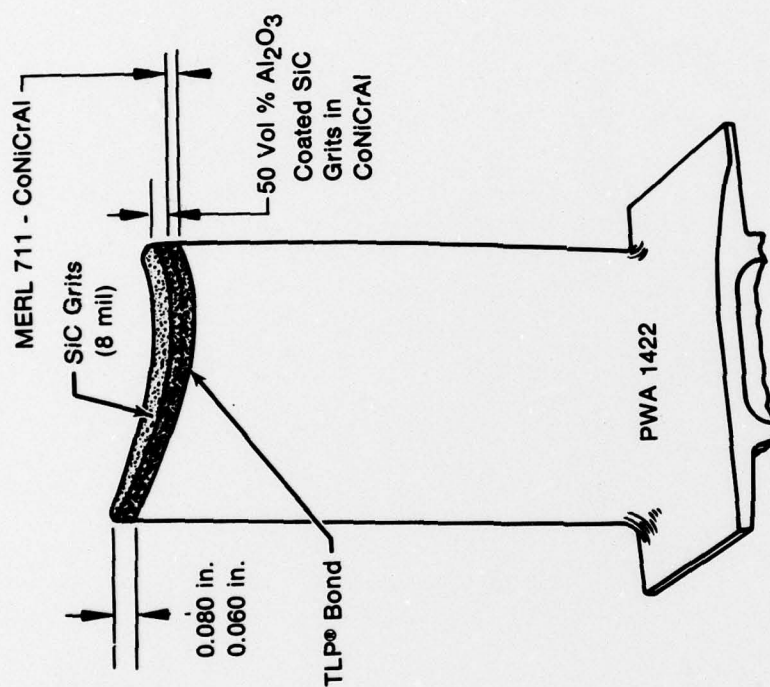
FD 111048

Figure 42. Rub Evaluation Comparison



FD 111049

Figure 43. Thermal Response of Interacted Blade Tips



FD 111060

Figure 44. Selected Blade Tip Treatment Coating